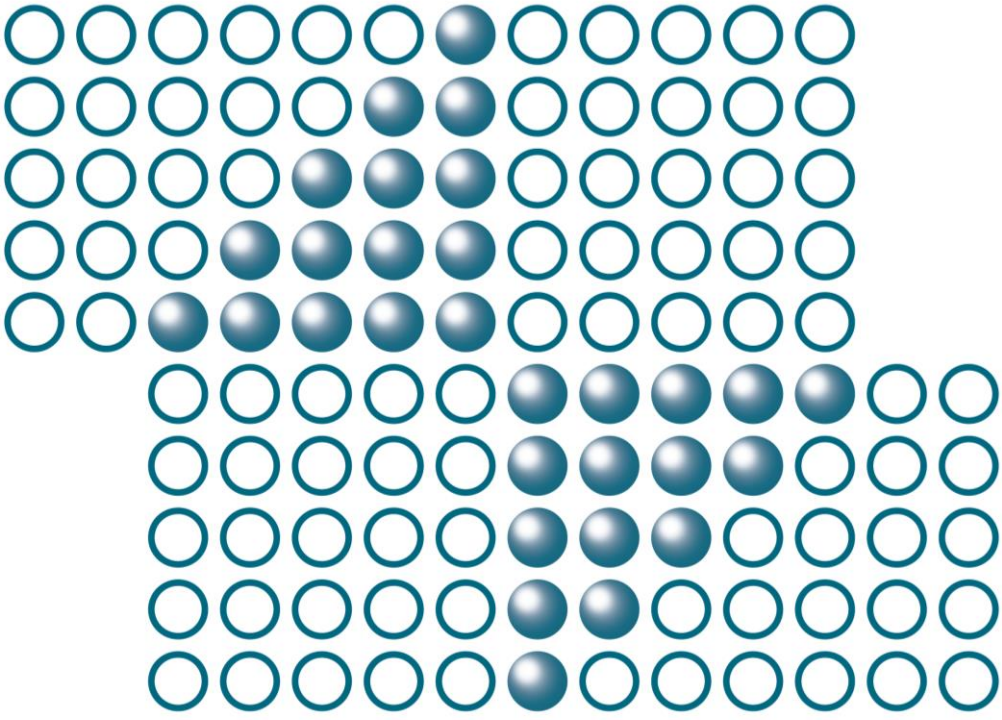
ABSTRACT BOOKLET

Abstracts of Contributions from the
19th International Symposium on Metallography,
Fractography and Materials Science

METALLOGRAPHY & FRACTOGRAPHY 2025



Edited by **Miloš MATVIJA and Peter HORŇAK**

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Abstracts of Contributions from the
19th International Symposium on Metallography,
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METALLOGRAPHY & FRACTOGRAPHY 2025

April 23 – 25, 2025

Hotel Atrium***
Nový Smokovec
High Tatra Mountains
Slovak Republic

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INVITED LECTURES

Effects of Gas Composition and Gas Pressure on Plasma Nitriding of Ferritic Stainless Steel Using 304 Stainless Steel Screen

Nishimoto Akio[1,a*], Tanaka Daichi[1] and Kishimoto Ojiro[2,b]

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Keywords: active screen; plasma nitriding; stainless steel; expanded ferrite; surface engineering.

Abstract

Plasma nitriding is an effective surface treatment that enhances hardness, wear resistance, and corrosion resistance, while offering advantages such as low energy consumption and short processing times. However, conventional plasma nitriding can cause defects due to direct plasma formation on the treated material. To mitigate this issue, the screen-assisted direct current plasma nitriding (S-DCPN) method was developed, generating plasma on both the treated sample and the screen to reduce defects. In this study, S-DCPN was applied to ferritic stainless steel (SUS430), using austenitic stainless steel (SUS304) as the screen material. The treatment was conducted at 633 K for 15 hours under gas pressures of 200 and 600 Pa, with varying gas compositions (75 % N_2 - 25 % H_2, 50 % N_2 - 50 % H_2, and 25 % N_2 - 75 % H_2). To investigate the effects of gas composition and gas pressure on the plasma nitriding of ferritic stainless steel, various analyses were conducted, including appearance observation, XRD analysis, surface microstructure observation, cross-sectional microstructure observation, glow discharge optical emission spectrometry (GD-OES), hardness testing, and corrosion testing. The results showed that the α_N phase, a supersaturated nitrogen solid solution, formed under all conditions. Nitrogen diffusion and hardness increased (Fig. 1), and corrosion resistance was enhanced, particularly under the 25 % N_2 - 75% H_2 gas composition (Fig. 2). These findings demonstrate the effectiveness of S-DCPN in improving surface properties without compromising corrosion resistance.

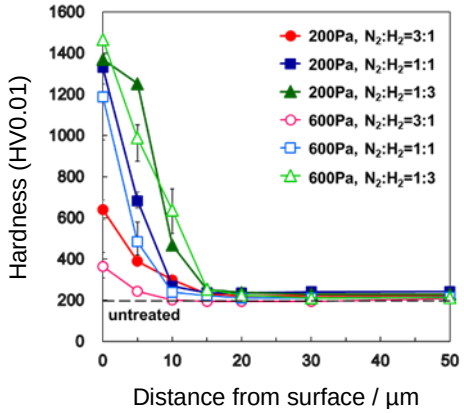

Fig. 1 Cross-sectional hardness of SUS430 sample treated by S-DCPN using SUS304 screen.

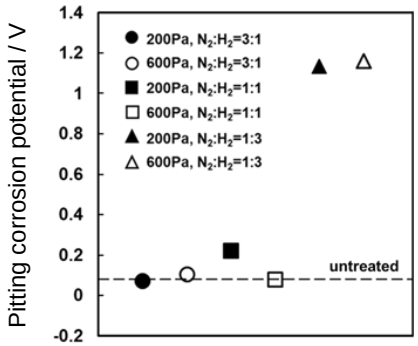

Fig. 2 Pitting potential of SUS430 samples treated by S-DCPN using SUS304 screen.

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Ceramography and Fractography in Dual-Phase High Entropy Ceramics Research

Dusza Ján

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Keywords: dual-phase high entropy ceramics; SEM; TEM; HRTEM; microstructure; fracture.

Abstract

A novel dual-phase high entropy composites were processed using different processing routes. Microstructure characteristics have been investigated at micro/nano/atomic scale based on the detailed SEM and STEM analyses, Fig. 1. Hardness and strength were investigated at micro/nano level, and the influence the microstructure characteristics on deformation mechanisms have been studied. Fractography was used for the characterization of fracture origins and fracture mechanisms.

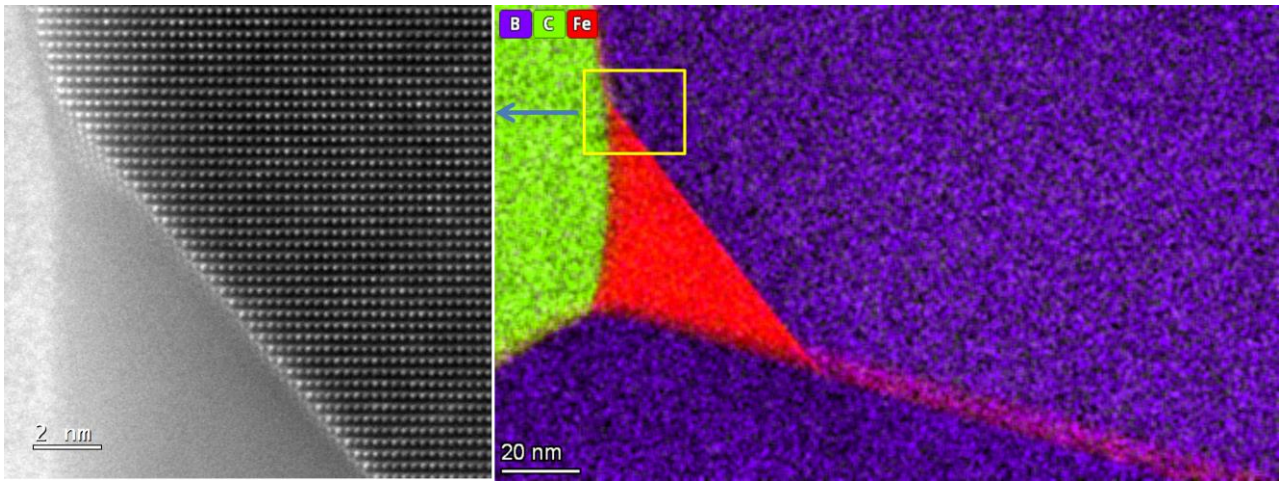

Fig. 1 Triple-point at boride / carbide grain boundary by HREM + EDAX.

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CONFERENCE LECTURES

Additively Manufactured and Heat Treated Dievear Tool Steel

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Keywords: powder bed fusion; tool steel; microstructure; mechanical properties.

Abstract

Dievar tool steel was prepared by laser powder bed fusion and subsequently heat treated. After printing, this steel reaches a tensile strength of 1870 MPa at 18 % ductility. Heat treatment of the steel was performed by quenching and one or two step tempering or tempering directly from the as-built condition. By tempering without prior hardening, the highest tensile strengths exceeding 2100 MPa were obtained after heat treatment.

Microstructure and Deformation Behavior of Ag-Cu Metastable Metal-Matrix Composite Prepared from Core-Shell Powder

Lejček Pavel[1,a*], Strakosova Angelina[1,b], Průša Filip[2,c] and Dvorský Drahomír[1,d]

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Keywords: metal-matrix composite; Ag@Cu core shell powder; plastic deformation; fracture.

Abstract

As metal-matrix composites (MMCs), the materials are usually considered, containing soft matrix and hard particulates of various shapes. Accordingly, they can well serve for numerous practical applications. From the viewpoint of understanding detailed mechanisms of deformation, it is also interesting to study MMCs containing not only soft matrix but also soft particulates. We refer to the microstructure and plastic deformation of Cu–Ag metastable MMCs (m-MMCs) produced by spark plasma sintering of the Cu@Ag core-shell powders. The microstructure is characterized by Ag matrix and Cu spherical particulates with the grain sizes of 2 μm and 11 μm, respectively. The true stress–true strain dependence of such m-MMCs can be phenomenologically described by the logarithmic dependence but also by quadratic functions. The parameters of these dependences are specified, quantified, and discussed with respect to those of pure Ag and Cu. Besides, the plastic deformation results in the prolongation of the particulates relative to the straining of the m-MMC, i.e., in the direction of tensile deformation and perpendicularly to the direction of compression. The aspect ratio of the particulates is related to the value of the strain, and it is shown that these values fit with a proposed model of sphere deformation [1].

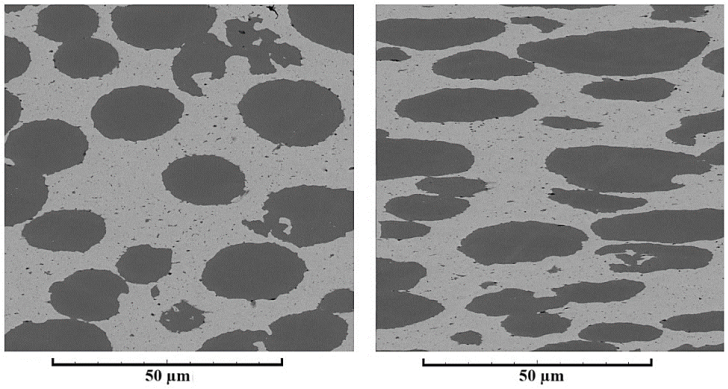

Fig. 1 SEM images of the cross-section parallel to the compression direction of 51:49 Ag–Cu m-MMCs produced by SPS: deformation by 23 % (left) and by 53 % (right). The axis of compressive deformation is in the vertical direction.

Acknowledgment

This work was financially supported by the Czech Science Foundation under the grant No. 23-05139S. AS acknowledges the grant of the Czech Academy of Sciences under No. L100102403.

Reference

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Microstructural Characteristics of Ultra-High Temperature Binary Carbides with Improved Mechanical Properties

Kovalčíková Alexandra[1,a*], Hrubovčáková Monika[1,b], Ďaková Lenka[1,c], Albov Dmitry[1,d], Hviščová Petra[1,e], Szabó Juraj[1,f], Sedlák Richard[1,g] and Csanádi Tamás[1,h]

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Keywords: ultra-high temperature ceramics; binary carbides; microstructure; nanohardness.

Abstract

This work is focused on the study of sintering conditions on microstructural characteristics and phase composition of high-temperature binary carbides. Transition metal elements of group IV and V of PTP were selected to prepare a combination of 12 binary carbides. The rationale to select these chemical compositions is due to higher thermal stability of their mono carbides, oxides or borides, known as UHTC ceramics.

Binary carbides, for example (TiZr)C, (ZrHf)C, (HfV)C, (VNb)C, (NbTa)C etc were spark plasma sintered at 2100°C for 10 or 20 minutes at 70MPa in argon atmosphere, respectively. To compare the resultant microstructure, 6 monocarbides TiC, ZrC, HfC, VC, NbC and TaC were synthetized at the same sintering procedure. Almost fully dense single phase carbides with porosity below 2% were prepared. However, the XRD confirmed the low amount of oxide phases as ZrO_2, TiO_2 or HfO_2. Longer time of sintering has not changed the grains size and porosity of binary carbides significantly. Nanohardness and Elastic modulus of experimental materials were also measured.

Acknowledgment

This work was supported by the Slovak Research and Development Agency under the contract APVV-22-0493, by Scientific grant agency VEGA 2/0107/24, and by the project M-ERA.NET 3/2021/295/Duplexcer.

Fine Tuning of Shape Memory Temperature in Metastable Ni2FeGa Heusler Alloys

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Keywords: Heusler alloy; Ni_2FeGa; shape memory effect; controlling of transformation temperature.

Abstract

The idea of shape memory temperature range controlling is based on the chemical composition varying of the alloy. For this purpose, Ni2FeGa Heusler alloy was selected as the best candidate from the point of applicability. This work is focused on the comparative study of the bulk alloy with the phases formed close to their equilibrium state and the microwires produced by rapid solidification by the modified Taylor-Ulitovsky method. The presented alloys were prepared with the chemical composition $Ni_{50}Fe_{25-x}Ga_{25+x}$ (x = -4, -2, 0, 2) which allowed the controlling of the transformation temperature range of the alloys from 100 K up to 400 K only in the form of microwires.

The combination of SEM/EDX/EBSD, micro-XRD analyses and microstructure observation provided the phase determination in both form of Heusler alloy and it was found that the microwires are formed in an oversaturated metastable state. In addition, non-equilibrium state stability of microwires was tested by cycling through the transformation temperature with no structural degradation and transformation temperature noted.

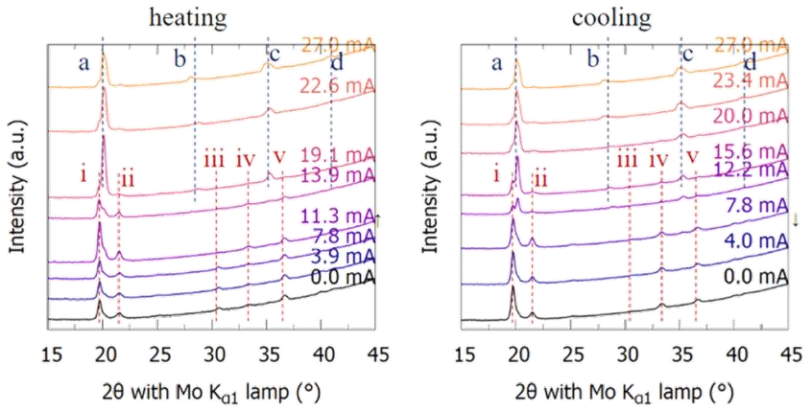

Fig. 1 Thermo-elastic martensitic transformation in $Ni_{50}Fe_{27}Ga_{23}$ microwire measured by micro-XRD and heated up by Joule method.

Acknowledgment

The authors were supported by the Scientific Grant Agency under contract VEGA projects No. 2/0086/22 and 1/0180/23.

The Influence of the Final Machining Process on the Change of the Microstructure in the Surface Region of Austenitic Stainless Steels

Bártová Katarína[1,a*], Kudláč Marek[1,b], Dománková Mária[1,c], Vopát Tomáš[1,d], Gavalec Matúš [1,e] and Slnek Dávid[1,f]

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Keywords: austenitic stainless steels; turning; microstructure; texture; corrosion.

Abstract

The corrosion resistance of austenitic stainless steels (ASSs) can be significantly degraded by improper state of the microstructure. Since the 1980s, stress corrosion cracking has been a serious degradation issue that threatens the reliability of components in nuclear power plants [1,2]. As some practical experience and subsequent studies show, stress corrosion cracking can also be initiated by the surface condition of the steel. Machining is usually the last stage of production, during which a highly deformed surface area with high residual tensile stresses can be created, which can accelerate the initiation of stress corrosion cracking [3]. This study is aimed at analysing the influence of final turning on the changes in the microstructural character of the surface-machined layer caused by various machining parameters (e.g.: cutting speed, feed rate, depth of cut, cutting tool geometry, etc.).

Observations confirmed significant changes in the microstructure in the surface area caused by the turning process. In particular, observations using the transmission electron microscope (TEM) on the thin lamellas prepared by the focused ion beam method confirmed the presence of a deformation texture at a depth of approximately 1 μm of the turned surface (Fig.1). Such the change of the microstructure can significantly affect the corrosion resistance of ASSs.

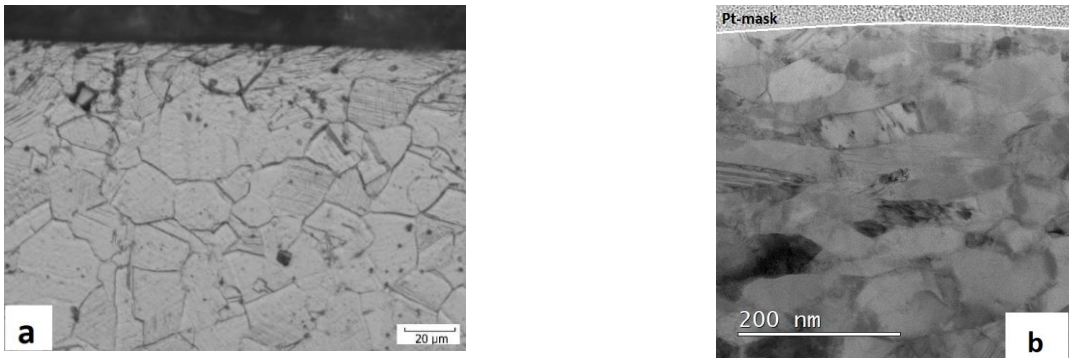

Fig. 1 Microstructure analysis of cross-sections of some samples using a) light optical microscopy, b) TEM.

Acknowledgment

This work was supported by the Slovak Research and Development Agency under the Contract no. APVV-22-0146.

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Novel ASC Refractory Design and Installation for Improved Energy Efficiency and Corrosion Resistance in Industrial Applications

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Keywords: Al_2O_3-SiC-C (ASC) refractories; corrosion resistance; industrial applications; energy efficiency.

Abstract

Al_2O_3-SiC-C (ASC) lining bricks for torpedo tank cars used in pre-treating molten (desulphurizaion) iron and molten iron ladles, which offer advantages such as high oxidation resistance, strong resistance to slag corrosion, good thermal shock resistance, and excellent resistance to mechanical scour wear and abrasion, have been investigated. It is expected that the combination of these new technologies will improve the energy and economic efficiency of the steel industry while also contributing to the decarbonization of both the refractory and steel industries. Additionally, the developed technology is expected to be applicable to other energy-intensive industries, such as cement, glass, pulp and paper, and non-ferrous metal processing.

Investigating post-mortem samples is crucial for reducing wear on ASC lining. The microstructures of post-mortem samples were analyzed using OLM, XRD, and SEM/EDS techniques. The results showed the formation of phases with low melting points, along with spinel solid solutions in the matrix and calcium dialuminate near the alumina aggregates. The results also indicated that slag and iron tend to react with alumina and silicon carbide, respectively. Molten slag causes refractory failure through corrosion dissolution and spalling.

Acknowledgement

This research was funded by Ministry of Education, Science, Research and Sport of the Slovak Republic: VEGA 1/0199/24: "Development of mathematical control models and digital twins for individual steel production processes based on machine learning with the aim of increasing the competitiveness of the sector and reducing the carbon footprint" and project 09I05-03-V02-00016: "Smart-Steel: AI-Driven control models for future steel production".

Active-Screen Plasma Nitriding of Small Thin Rolled Stainless Steel Plates

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Keywords: austenitic stainless steel; active screen plasma nitriding; expanded austenite; small thin rolled plate.

Abstract

This study aims to clarify the mechanical properties of expanded austenite (γ_N phase) formed in austenitic stainless steel (ASS). A small, thin-rolled plate of 304 and 316L stainless steels with a thickness of 0.5 mm was used as the test sample (Fig. 1). The sample was nitrided using active screen plasma nitriding (ASPN) at low processing temperatures of 400 °C and 450 °C for a duration of 4 hours. The γ_N phase was successfully formed on the sample surface as shown in Fig. 2.

Figure 3 shows the stress-strain diagram in a tensile test. In the SUS304 sample, the ASPN-treated sample had improved tensile strength and significantly reduced breaking elongation compared to the untreated sample in Fig. 3 (a). In the SUS316L sample, the ASPN-treated sample also showed improved tensile strength and reduced breaking elongation compared to the untreated sample in Fig. 3 (b), but these improvements were not as great as those for SUS304.

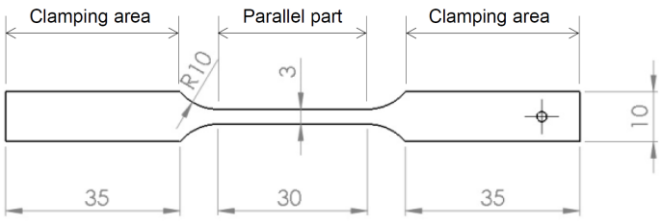


Fig. 1 Shape of a test sample of small thin rolled plate.

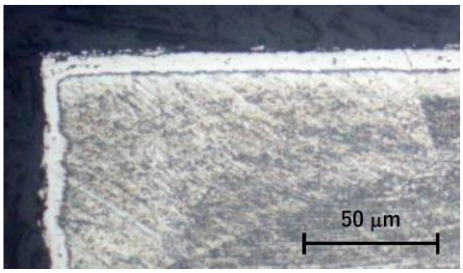
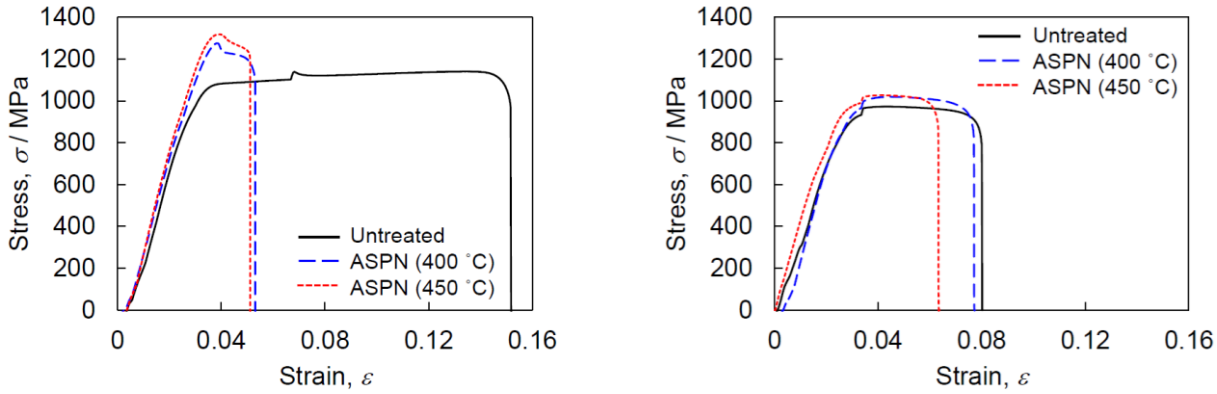


Fig. 2 Micrographs of ASPN sample (SUS304).



(a) SUS304 (b) SUS316L

Fig. 3 Stress-strain diagram of ASPN-treated samples.

Roman Bronze Objects from the Archaeological Site of Burg in Burgenland, Austria

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Keywords: bronze; small parts; Roman; microstructures.

Abstract

Several Roman bronze objects were confiscated from a digger, which had been collected illegally at the archaeological site of Burg, Burgenland. Since these parts are archaeologically worthless, they were allowed to be examined with destructive analysis methods. The investigative results of three parts, a hook, a fibula catch, a fibula head, are presented (Fig. 1).

The surface of the parts is covered with a green patina which contains mainly Cu and smaller amounts of Sn, Pb, P, Ca, Al, S and Fe.

The average XRF analyses of the hook showed a content of 0.8 wt. % Sn and about 2 wt. % Pb, but in the fibular parts approximately 7.5 wt. % Sn and 23 wt. % Pb were detected. In these analyses, it must be taken into account that elements such as Sn accumulate in the patina.

Due to the very different alloy components in the samples, the microstructures are also appropriate different [1, 2]. It is also possible to distinguish between cast and deformed microstructures. These investigations show that the Roman metallurgist used a wide variety of copper alloys, because raw and recycled materials were probably processed together.

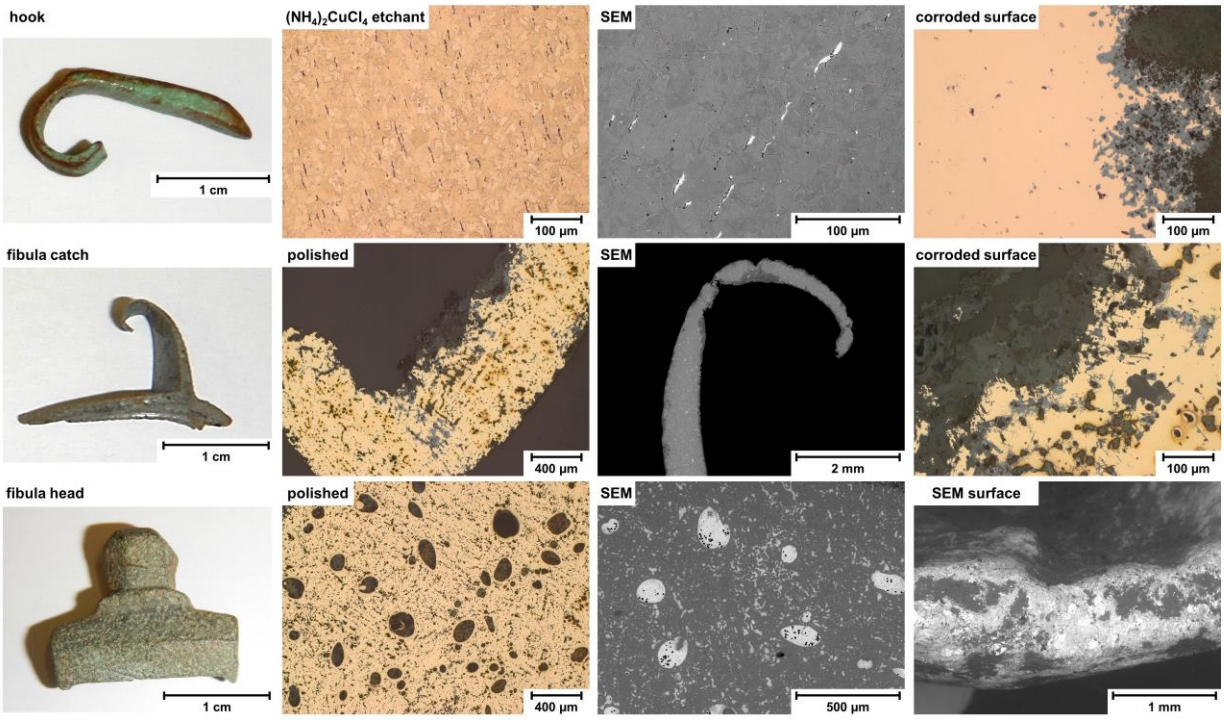

Fig. 1 Three small bronze parts with microstructure images in LOM and SEM.

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A Bronze Button from Late Bronze Age Site of Inzersdorf ob der Traisen

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Keywords: bronze; button; bronze age; ancient bronze ingot.

Abstract

In the region of Inzersdorf ob der Traisen in Lower Austria, 273 cremation graves from the late Bronze Age (ca. 1300 – 800 BC) were recovered. At this site some bronze objects were found, among them garment trimmings [1]. One of these objects is a button which was available for investigations. It should be determined how the button was manufactured, for example by casting or soldering, and microstructural changes can be detected due to temperature effects by cremation [2].

The button is made of bronze whose composition was determined by XRF: 87 wt. % Cu, 9 wt. % Sn and 1 wt. % Pb. The microstructure is rather homogeneous in the area of the eyelet but some parts of the plate have shrinkholes. It is possible that these areas melted during cremation (Fig. 1).

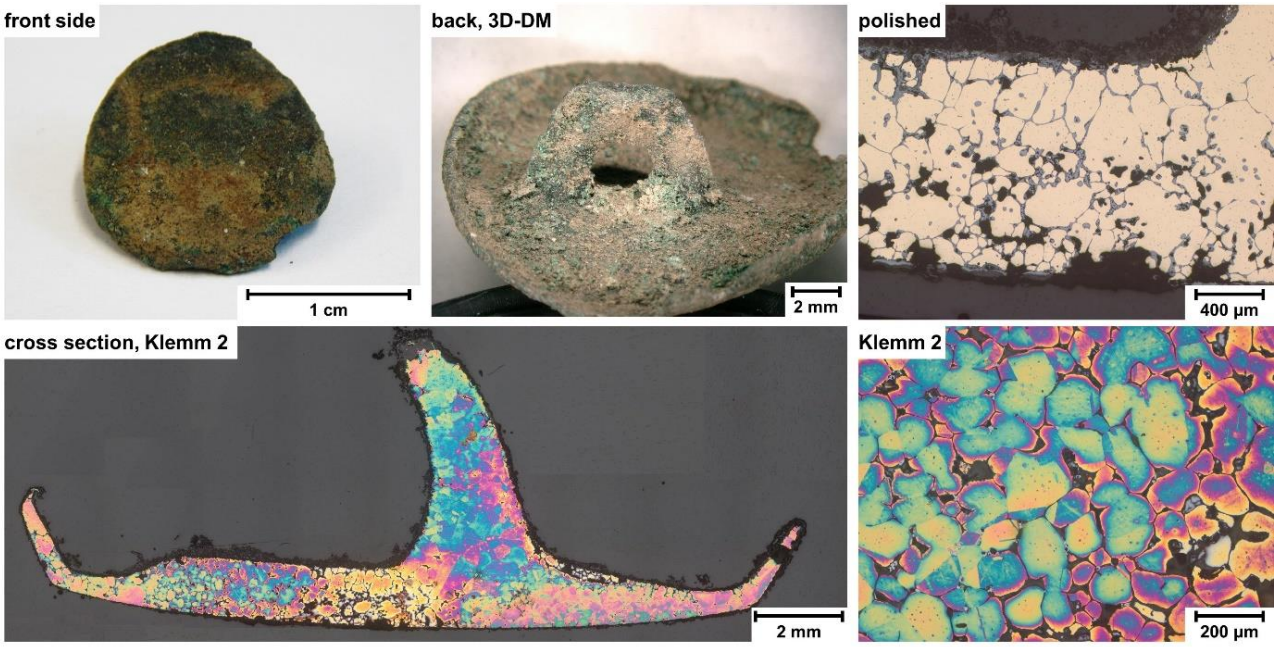

Fig. 1 Button from Inzersdorf ob der Traisen. Fotos and microstructures after metallographic preparation.

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Copper Test Melts with Additions of Pb, Bi, As, Sb and Sn

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Keywords: copper; test melts; bismuth; arsenic; antimony.

Abstract

Archaeometallurgical copper-artefacts contain a wide variety of metal admixtures (e.g. Pb, Bi, As, Sb, Sn) which either originate from the ores or were intentionally added [1, 2, 3]. When the melt solidifies, these elements can accumulate in different structural areas and form special phases. The different alloying elements also interact with each other.

In order to be able to examine these interactions, model alloys with different elements and concentrations were produced. These alloys were then prepared by metallography and examined using LOM and SEM (Fig. 1).

More simple alloys show a dendritic microstructure and the added elements accumulate in the interdendritic areas. This is clearly visible for Pb and Bi additions, as both metals are not soluble in copper.

On the other hand, As and Sb form compounds with Cu which precipitate. On the other hand, Sn is soluble in Cu at lower concentrations and Cu-Sn phases were formed only at higher concentrations.

The resulting structures become more complex if more elements are added, but they enable us for a better understanding the structures of ancient copper alloys.

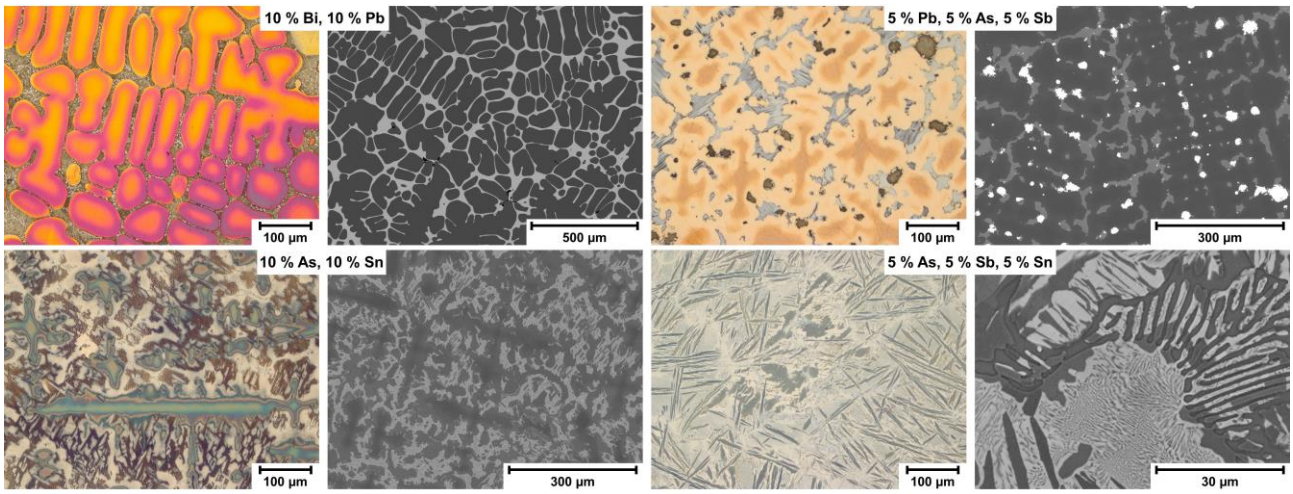

Fig. 1 Microstructure of four selected alloys in LOM and SEM.

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Enhancement of Mechanical Properties in $Al_{0.35}CoCrFeNi$ Complex Concentrated Alloys Through Grain Size Tailoring

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Keywords: complex concentrated alloy; recrystallization; grain growth; activation energy; Hall-Petch relationship.

Abstract

This research presents experimental results on the processing of CCA with a nominal composition of $Al_{0.35}CoCrFeNi$. The alloy was produced by vacuum induction melting and tilt casting. The microstructure of the as-cast CCA consists of dendritic and interdendritic regions homogenized by heat treatment at 1360 °C. After rotary swaging at room temperature, the microstructure is characterized by an abundance of dislocations and continuously intersecting slip bands. Annealing experiments were carried out in the temperature range of 1150 °C – 1300 °C and different holding times to determine the parameters of grain growth kinetics. Phase and chemical analysis were investigated using XRD and EDS methods.

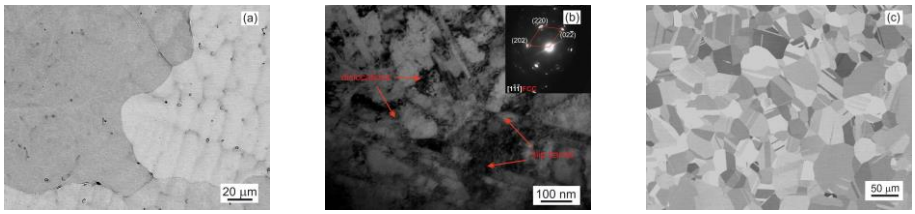

Fig. 1 The typical microstructure of the $Al_{0.35}CoCrFeNi$ CCA: (a) SEM micrograph of the as-cast dendritic microstructure; (b) TEM bright-field images of the swaged alloy with corresponding SADP; (c) SEM micrographs of annealed alloy at 1150 °C for 3 min.

The activation energy of recrystallization in the studied composition was 458 kJ mol^{-1} [1]. The influence of grain size on room temperature mechanical properties and tensile properties was determined. It was found that relative elongation decreases somewhat with decreasing annealing time. However, the alloy still demonstrates high plasticity (≥50 %). At the same time, the values of UTS and YS increased with decreasing annealing time and amounted to 705 MPa and 291 MPa, respectively. The hardening coefficients k_h and k_σ, calculated using the Hall-Petch relation, were 277.5 HV $\mu m^{-1/2}$ and 655 MPa $\mu m^{-1/2}$ respectively, indicating the effectiveness of grain boundary hardening in the studied single-phase CCA.

Acknowledgment

The Slovak Research and Development Agency financially supported this work under the contracts APVV-20-0505 and the Slovak Grant Agency for Science under VEGA 2/0018/22. The EU NextGenerationEU also Funded this work through the Recovery and Resilience Plan for Slovakia under project No. 09I03-03-V01- 00037.

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Impact of Primary Piping Material Conditions on Safe Long-Term Operation of VVER-Type NPPs: Delisa-LTO project

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Keywords: nuclear power plant; long-term operation; VVER-440; thermal aging.

Abstract

The extension of the original operational lifetime and the long-term operation (LTO) of nuclear power plant (NPP) units depend on the compliance with strict safety standards. In order to ensure continuous safe operation of world reactors, it is necessary to solve specific issues affecting the renewal of LTO licenses. The decommissioning of two VVER-440 units in Slovakia brings a great opportunity to obtain materials from equipment that was in conditions of real operation for decades (app. 28 - 29 campaigns) and became the main source of experimental material for the European HORIZON-EURATOM project DELISA-LTO. Its primary goal is to test and study thermal ageing effect, which is one of the main degradation processes occurring in primary circuit components. By utilizing experimental techniques such as mechanical testing and NDT inspection methods, researchers hope to gain a better understanding of this material degradation phenomenon.

The aim of this paper is to describe the preliminary results from optical microscopy. The paper also provides overview of the experimental background that has been used to create a comprehensive roadmap starting from the material selection, cutting plans, distribution to partners and preparation for the actual experimental testing.

Acknowledgment

This paper was support by the DELISA-LTO project n°101061201 funded by the European Union.

Evaluation of Microstructure Changes in Alloy F (Cr-Ni) and Inconel A560 Materials before and after Annealing

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Keywords: microstructure; Inconel A560; melting pot; heat-treating; temperature cycles.

Abstract

The article focuses on the evaluation of changes in the microstructure of two materials used in the production of a melting pot for glass remelting. The alloys Cr-Ni – F (original supplier) and Inconel A560 (new supplier) are compared – before, and after heat-treating process, which was not performed by the new manufacturer, although it was recommended. The Inconel A560 alloy was chosen as a replacement for the original alloy F. The experiment was motivated by problems with the low durability of melting pots, which did not meet the requirements for the number of temperature cycles.

The analysis showed that the material of the new pots has a heterogeneous structure, with segregation of different phases at the grain boundaries in the interdendritic region. This phenomenon can affect the mechanical properties and reduce the service life of the material. The findings indicate that performing the recommended heat-treating process could improve the homogeneity of the structure and the resistance of the material to thermal cycling. The article emphasizes the importance of adhering to technological procedures and quality control in the production of the melting pots from Inconel A560 material.

The Impact of Final Turning on the SCC Susceptibility of Austenitic Stainless Steel AISI 304 and AISI 321

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Keywords: austenitic stainless steel; final turning; stress corrosion cracking; corrosion; surface.

Abstract

Final turning, which is a finishing process for obtaining components with specific precise parameters, affects the integrity of the surface and its properties, whether hardness or surface residual stresses. The synergistic effect of these factors affects the susceptibility of the material, to stress corrosion cracking.

In this work, 2 types of austenitic stainless steel, namely AISI 304 and AISI 321, were turned. These steels are a frequent research material in terms of stress corrosion cracking as they are widely used in the primary circuits of nuclear power plants, where such manifestations of corrosion degradation occur very frequently. Regarding machining, a tool with positive cutting geometry was used. The cutting parameters that varied were the cutting speed (100 and 250 m min^{-1}) and the tool feed (0.12, 0.2 and 0.3 mm). The depth of cut was the same for all turnings (0.8 mm).

Subsequently, the prepared samples were exposed in $MgCl_2$ solution based on the ASTM G36. This test provides an accelerated determination of SCC susceptibility precisely due to this extremely aggressive environment. The exposure test was carried out for 96 hours. After this time, the samples were analyzed using SEM, where the density of surface cracks was monitored. When comparing the crack density, an increase in density was visible for AISI 304 compared to AISI 321. It was shown that with increasing cutting speed, the density of cracks increased significantly, as well as with increasing tool feed. On the cross-sections the depth and length of the cracks were analyzed. Crack depth and length increased with increasing feed too.

Acknowledgment

This work was supported by the Slovak Research and Development Agency under the Contract no. APVV-22-0146. This work was supported by the call for doctoral students and young researchers of Slovak University of Technology in Bratislava to start a research career (Grant 23-06-09-A).

New Approaches for Evaluating the Resistance of Clads under High Temperature Corrosion Conditions

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Keywords: high-pressure casting; cladding; PVD nanocoating; high temperature corrosion.

Abstract

The aim of the experimental work was to propose innovative procedures for the formation of renovation layers, to determine suitable material, modify the microgeometry and surface topography of new and renovated shaped parts of moulds for high-pressure casting of aluminium alloys. It has been designed and validated under laboratory and operational conditions a method of modifying the surface of the mould parts of moulds for casting aluminium alloys by forming stochastic texture by low energy laser in combination with duplex PVD coatings on the surfaces of mould parts in contact with the aluminium alloy melt [1, 2]. It has been verified the contact angle measurement methodology for determining the number of spurs by separation lubricant on the surface of the new or refurbished mould part before the first casting cycle. Tribological information of surfaces with laser textured surface and deposited PVD coating dupex NACRo4 were determined by measuring the coefficient of friction by the Pin-on-Disc method. For the formation of the renovation layers, the additive materials were verified Dievar, Dratec, UTPA 702. A TruDisk 4002 solid-state disk laser with BEO D70 focusing optics was used for winding. For the purpose of determining the resistance to high temperature corrosion of the coating and the base material (BM), the corrosion resistance coefficient was defined as the ratio of the thickness of the intermetallic layer formed on the surface of the coating to the thickness of the intermetallic layer formed on the surface of the base steel material, Fig.1.

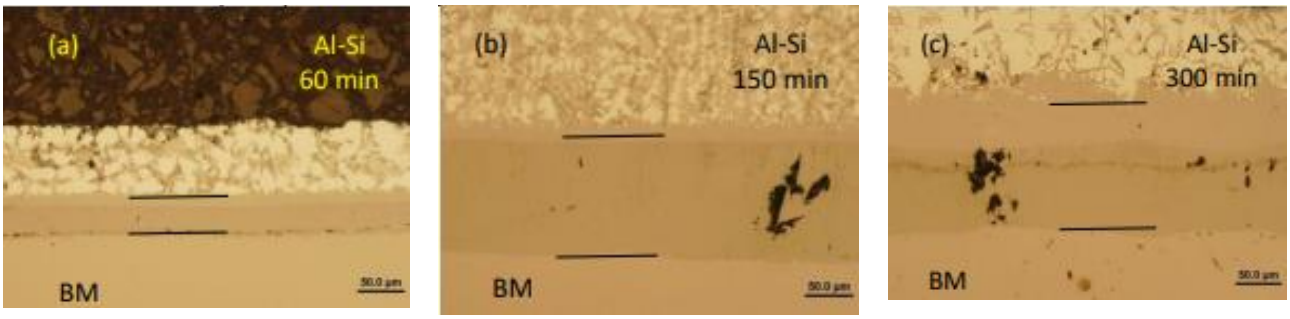

Fig. 1 Changes in the intermetallic layer after immersion in an Al-Si based aluminium alloy melt.

Acknowledgment

This research was funded with the support of VEGA 1/0597/23, APVV-20-0303 and KEGA 024TUKE-4/2025.

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Hardness Anisotropy of HfC and TaC Ceramic Grains

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Keywords: hardness anisotropy; slip systems; transition metal carbides; nanoindentation.

Abstract

The anisotropy of hardness and indentation modulus of grains of low-index crystallographic orientations ({001}, {101} and {111}), mapped by electron backscatter diffraction, were investigated by nanoindentation in polycrystalline HfC and TaC ceramics. It was revealed that the hardness anisotropy exhibited different trends for the HfC and TaC ceramics while the indentation modulus did not show detectable anisotropy. The {101} and {111} facets of HfC is found to be harder (~32 GPa) than the {001} orientation (~30 GPa) while, in the case of TaC, higher hardness corresponds to the {111} facet (~23 GPa) in comparison with the {001} and {101} orientations (~22 GPa) as shown in Figure 1. The different hardness anisotropies are attributed to the different dominant slip activation reported in the literature, providing a quick and efficient tool for the distinction between the <1-10>{110} and <1-10>{111} type active slip systems in rock salt structure transition metal carbides.

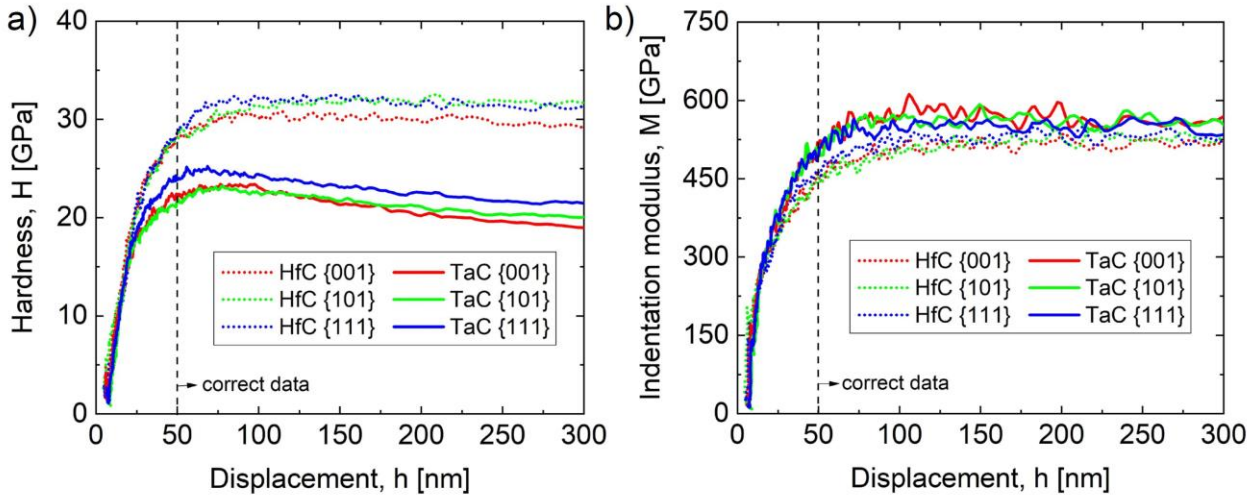

Fig. 1 Typical a) hardness – and b) indentation modulus – depth curves of HfC and TaC grains measured on low index crystal facets.

Acknowledgment

This research was supported by the Slovak Grant Agency for Science via the projects APVV-19-0497 and APVV-22-0493 and by the Slovak Academy of Sciences via the projects: Seal of Excellence – Strengthecs and IMPULZ IM-2022-67.

Impact of Dual-Beam Laser Welding Energy Distribution on the Structural and Mechanical Properties of DSS 2304 Weld Joints

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Keywords: dual laser beam welding; mechanical properties; ferrite-austenite ratio.

Abstract

This study investigates weld joints in duplex stainless steel DSS 2304 (Table 1), fabricated using dual-beam laser welding with consistent parameters but varying ratios of energy distribution between the beams. Duplex stainless steels have a widespread application in marine environments, heat exchangers, and chemical industries. Typically, these steels consist of approximately equal parts austenite and ferrite, combining the strength of ferrite with the ductility and toughness of austenite to offer excellent corrosion resistance.

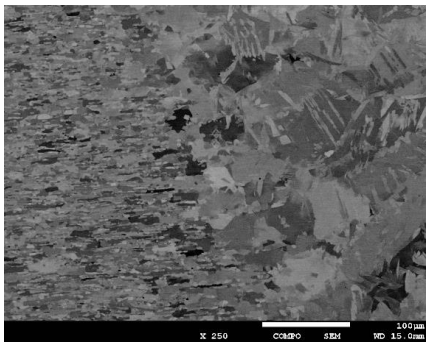
The welding process plays a crucial role due to its direct influence on the material's microstructure and resulting properties according One of the primary challenges in welding duplex steels is achieving a balanced ferrite-austenite ratio in the weld metal without requiring post-weld heat treatment. This article explores how different energy distribution ratios in dual-beam laser welding affect the structural and mechanical characteristics of weld joints in 2mm-thick DSS 2304.

The study involves macroanalysis to assess weld geometry and average width, microstructural analysis to examine the ferrite-austenite ratio and grain size across weld regions, and evaluation of mechanical properties through microhardness and tensile strength testing, with additional analysis of fracture surfaces post-testing. The findings are compared and analyzed to determine the impact of energy distribution ratios on weld joint properties.

Table 1 Chemical composition of DSS 2304 [wt. %]

C	Mn	Si	P	S	Cr	Mo	Ni	Cu	N
0.02	1.37	0.63	0.03	0.001	23.80	0.55	4.30	0.28	0.19

a)



b)

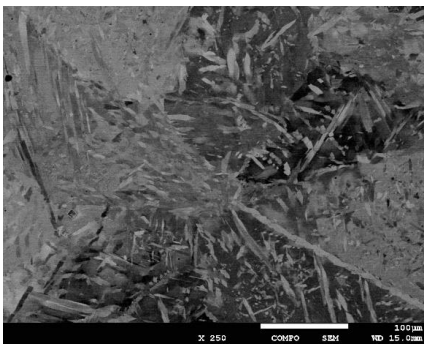


Fig. 1 Microstructure of DSS 2304 with BSE: a) Heat affected zone, b) Fusion zone.

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Evaluation of Interface between Alumina Dispersion Strengthened Copper Composite and Precipitation Strengthened Copper

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Keywords: alumina dispersion strengthened copper; spark plasma sintering; spot welding; copper alloy.

Abstract

Alumina-dispersed copper composites (ADSC) are materials with excellent thermal stability, electrical and thermal conductivity, and relatively high hardness compared to pure copper. By introducing only 1% Al_2O_3, it is possible to produce a composite exhibiting a hardness of 115 HBW and a yield strength of 288 MPa, with minimal changes even when exposed to temperatures up to 900 °C [1]. These properties make ADSC a promising material for high-temperature applications where electrical conductivity is required. One such potential application is in welding electrode tips, which experience high stresses at elevated temperatures. The preparation of alumina-dispersion-strengthened materials is predominantly achieved through powder metallurgy which allows homogeneous distribution of dispersoids in composite. In this study, Spark Plasma Sintering (SPS) was used for compaction, as it enables the production of highly dense compacts with isotropic mechanical properties. However, despite its advantages, the SPS method has certain technological limitations, such as the relatively small sample size due to the design constraints of SPS machines. To overcome these limitations, it is necessary to explore methods for joining ADSC with commercially used precipitation-strengthened copper to achieve sufficient dimensions for practical welding applications. During spot welding, the temperature at the contact surface between the electrode and the welded material can exceed 900 °C [2], causing a decrease in the hardness and strength of the CuCr precipitation-strengthened alloy. A potential solution is to manufacture the contact surface – primarily affected by heat and abrasion – using highly resistant ADSC, while the rest of the electrode body is made from a less stable but more cost-effective material. The aim of this study is to evaluate different methods of joining ADSC with precipitation-strengthened copper. Three approaches: simple mechanical joining, welding via SPS, and in situ compaction of ADSC will be compared. The advantages and disadvantages of each method will be analyzed and discussed.

Acknowledgment

This work was carried out within the research project VEGA 2/0092/24.

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Polycrystalline Thermoelectric Materials with Observed Anisotropy

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Keywords: thermoelectric generator; SnSe; Ag_2S; anisotropy; doping.

Abstract

Thermoelectric generators (TEGs) are highly reliable and predictable energy sources, particularly vital in extreme environments such as deep space exploration and off-grid terrestrial applications. However, their operational longevity can be compromised by mechanical stresses arising from thermal expansion mismatches between dissimilar thermoelectric materials in conventional heterojunction couples. Tin selenide (SnSe) has emerged as a promising candidate for homojunction TEG pairs. Dopant selection critically influences both thermoelectric and mechanical performance of this material. Our Bi-doped polycrystalline SnSe materials demonstrate a unique polarity switching phenomenon, transitioning from p-type to n-type conduction at specific dopant concentrations (>0.5 at. %), while maintaining comparable properties to the original SnSe. This work additionally investigates the anisotropic behavior of polycrystalline SnSe doped with Bi, Ag, Ni, Mg, and In, synthesized via scalable powder metallurgy and spark plasma sintering (SPS) techniques. The resulting materials exhibit a layered microstructure with pronounced preferred orientation, leading to anisotropic properties that are further amplified during sintering.

This work also explores ductile Ag_2S materials, which combine thermoelectric performance with exceptional ductility not common for thermoelectrics. These materials exhibit fast thermal response times and high temperature sensitivity, making them ideal for wearable energy harvesters and biocompatible medical sensors capable of continuous physiological monitoring

By optimizing SnSe homojunctions through anisotropic engineering and dopant selection, this work enables TEG designs with reduced interfacial stresses and enhanced durability without sacrificing performance. When combined with flexible Ag_2S systems, it opens new possibilities for hybrid thermoelectric devices operating across wide range of small and large temperature gradients in both aerospace and biomedical applications.

Acknowledgment

This work was supported by the Slovak Research and Development Agency under Contract no. APVV-23-0030, VEGA project No. 2/0039/23 and KEGA project No. 011TUKE-4/2025. This research was funded in part by the international project M-ERA.NET 3/2022/235/H2MobilHydride.

POSTER PRESENTATIONS

Effects of Ball Milling Time on CoCrFeNiTi High Entropy Sintered Alloys

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Keywords: high entropy alloy; spark plasma sintering; mechanical alloying; powder metallurgy.

Abstract

High-entropy alloys (HEAs) are known for their excellent mechanical properties and thermal stability, achieved by combining five or more metallic elements in nearly equiatomic proportions [1]. In our laboratory, we are aiming to fabricate uniform HEAs by employing a process that combines mechanical alloying (MA) and spark plasma sintering (SPS), which is particularly effective for alloying high-melting-point metals and those with significant differences in melting points and densities. In previous studies [2], a CoCrFeNiTi-based HEA was produced using the MA-SPS process, where a sintered body was obtained from the alloy powder; however, it was found that carbon contamination – presumed to originate from the solvent used during MA – was present.

In the present study, Co, Cr, Fe, Ni, and Ti powders (particle size <45 μm, purity >99.5 %) were processed with varying MA times (0, 5, 10, 15, 25, and 50 hours) to produce homogeneous alloy powders and fabricate sintered bodies. A series of tests were then conducted to thoroughly evaluate the origin and effects of the contamination, as well as to investigate the changes in mechanical properties and crystal structure under varying MA times.

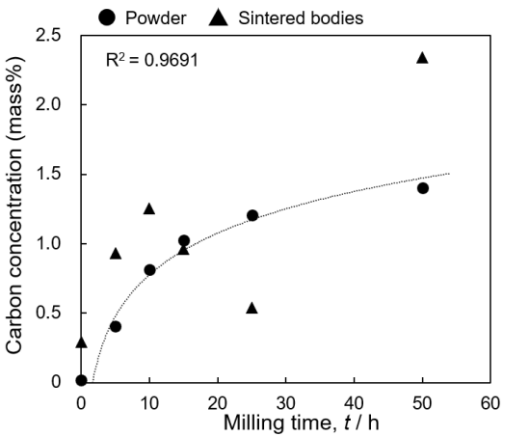

Fig. 1 Results of carbon concentration for powder and sintered body by EMIA and EPMA.

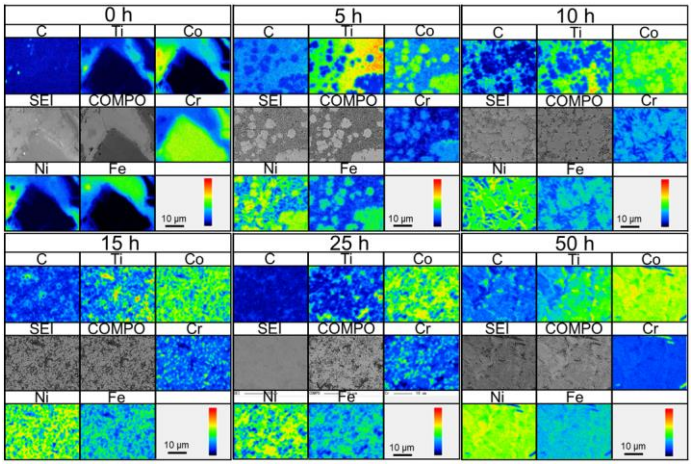

Fig. 2 Results of qualitative analysis of sintered bodies for each BM time.

Fig. 1 shows that the carbon concentration in the powder increases logarithmically with MA time. This suggests that the plastic deformation energy accumulated during mechanical alloying is released, triggering a mechano-chemical reaction of heptane that leads to carbide formation. In addition, as illustrated in Fig. 2, increasing the MA time results in a more uniform distribution of Ti and C in the sintered bodies, although a localized enrichment of Cr also appears concurrently.

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High-Entropy Alloy AlCoCrFeNi2 and its Heat Treatment

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Keywords: high-entropy alloy, high-temperature, microstructure.

Abstract

During the experiment, the properties of a high-entropy alloy (HEA) AlCoCrFeNi2 were analyzed. This alloy was designed for high-temperature applications, so its high-temperature properties were tested. The alloy was cast in a mould; after casting, it consisted of two phases (both cubic crystal lattice, one phase enriched in Al and Ni, the other phase enriched in Cr, Co, Fe) and exhibited a dendritic structure, as shown in Fig. *1*.

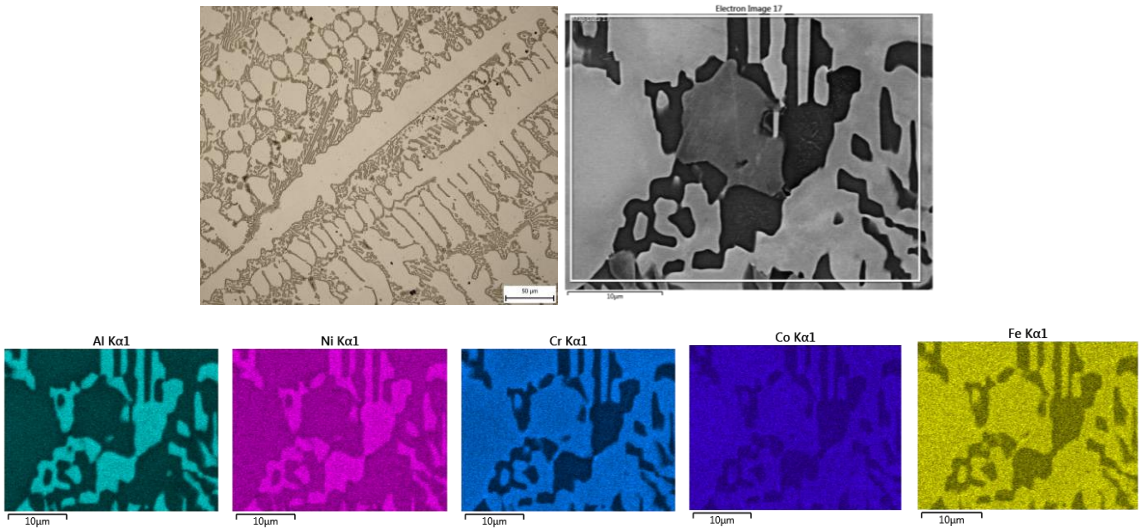

Fig. 1 Microstructure (light microscopy) and EDS (selective electron microscopy).

Subsequently, the alloy was exposed to several types of heat treatment: 1) short-term: at 950 - 1200 °C (30 min) - higher temperatures led to a change in microstructure (at tested temperatures, segregation appeared in the interdendritic spaces, at 950 °C it was highest and gradually decreased with increasing temperature) and a slight decrease in hardness (from 264 to 247 HV10), the effect of the cooling medium was also investigated, and it was observed that higher hardnesses are achieved in the case of cooling in air compared to water (opposite result compared to steel quenching), 2) medium-term: 650 - 1200 °C (8 h and 24 h) - higher temperatures led to a change in microstructure and a decrease in hardness, the dwell time did not affect the microstructure or hardness, 3) long-term: 1280 °C (24, 48, 72 and 96 h) - a slight increase in hardness with a longer residence time. After all heat treatment regimes, the material still consisted of the same two phases. Disruption of the dendritic structure occurred only at 1280 °C.

Acknowledgment

The present contribution has been prepared with the support of project TH82020002 Development and processing of advanced metal hydride composites with specific microstructural properties for hydrogen storage in mobile applications. The project was funded from specific state budget resources for research and development.

Design and Characterization of High-Entropy Alloys for Hydrogen Storage

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Keywords: hydrogen sorption; metal hydrides; hydrogen affinity; high-entropy alloys.

Abstract

This study introduces an innovative approach to alloy design based on the semi-empirical model developed by Griessen and Driessen, which predicts an alloy's hydrogen affinity [1]. Four equimolar HEAs forming a single phase were designed and synthesized with compositions favouring low hydrogen affinity due to their highly endothermic enthalpies. The prepared samples were subjected to a complete characterization of their material properties. The powder materials were used for the determination of the hydrogen sorption properties. According to the prediction model, these samples were not expected to form hydrides. Experimental results confirmed negligible hydrogen absorption, with a maximum of 0.23 wt. % (H/M = 0.13). This work highlights the significance of the semi-empirical model of Griessen and Driessen in tailoring alloys for specific applications, such as maximizing hydrogen storage or minimizing hydrogen absorption in materials susceptible to hydrogen embrittlement.

Table 1 Properties of the investigated HEAs

Alloy *EDX composition* [at.%]	**Density** [g.cm^{-3}]	**Hardness** HV0.3	**XRD**	**Temperature of max. H_2 Absorption [°C]**	**Maximum H_2 Capacity [wt.%]** *(H/M)*
HEA1: CoNiMnCrFe $Co_{21}Ni_{20}Mn_{16}Cr_{22}Fe_{21}$	7.99	130±5	FCC	175	**0.23** *(0.13)*
HEA2: CoNiMnCrCu $Co_{21}Ni_{20}Mn_{18}Cr_{20}Cu_{21}$	7.81	220±4	FCC	125	**0.14** *(0.08)*
HEA3: CoNiMnFeCu $Co_{21}Ni_{21}Mn_{17}Fe_{21}Cu_{20}$	7.99	170±5	FCC	RT	**0.08** *(0.04)*
HEA4: CoNiAlCrFe $Co_{20}Ni_{20}Al_{20}Cr_{20}Fe_{20}$	7.06	492±11	BCC	RT	**0** *(0)*

Acknowledgment

This work was funded by the Slovak Research and Development Agency (APVV-20-0205; APVV-21-0274), the Scientific Grant Agency of the Ministry of Education, Science, Research and Sport of the Slovak Republic and the Slovak Academy of Sciences (VEGA project No. 1/0122/25), and the international projects EIG CONCERT-JAPAN/2021/215/EHSAL and M-ERA.NET 3/2022/235/H2MobilHydride.

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Exploring Metal Hydrides for Efficient Hydrogen Storage

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Keywords: hydrogen storage; metal hydrides; alloy design; energy materials; high-entropy alloys.

Abstract

Achieving a sustainable and environmentally friendly energy landscape requires the development of innovative materials capable of efficient hydrogen storage. Among various storage solutions, metal hydrides represent a promising class of materials due to their ability to reversibly absorb and release hydrogen under controlled conditions. Their high volumetric hydrogen density, tunable thermodynamic properties, and potential for safe and compact storage make them attractive candidates for energy applications. However, optimizing their performance requires a deeper understanding of the structural and compositional factors influencing hydrogenation behavior.

Recent advancement in alloy design have enabled the development of medium-entropy and high-entropy alloys, which exhibit favorable thermodynamic and kinetic properties for hydrogen uptake. These materials offer unique structural characteristics, including lattice distortions and compositional complexity, which can influence hydrogen solubility, diffusion, and stability. Investigation of the interplay between atomic-scale features and hydrogen absorption/desorption mechanism is crucial for tailoring their properties to practical applications.

This study focuses on the influence of compositional variations and microstructural characteristics on the hydrogen storage performance of selected alloys. By employing advanced characterization techniques, insights into the phase stability, hydrogenation kinetics, and the decomposition behavior of hydrides during desorption under controlled conditions are obtained. A combination of in-situ and ex-situ analytical methods provides a comprehensive understanding of the structural transformations occurring during hydrogen release. The findings contribute to the broader scientific knowledge of hydrogen storage in complex metal hydrides and support the development of next-generation materials for efficient and sustainable hydrogen-based energy systems.

Acknowledgment

This work was supported by the Slovak Research and Development Agency under the Contract no. APVV-21-0274 and APVV-20-0205. Authors are grateful to the Scientific Grant Agency of the Ministry of Education, Science, Research and Sport of the Slovak Republic and the Slovak Academy of Sciences VEGA project No. 1/0122/25. This research was funded in part by the international projects M-ERA.NET 3/2022/235/H2MobilHydride and EIG CONCERT-Japan/2021/215/EHSAL.

Ablation Resistance of High-Entropy Carbide Ceramics with the Addition of SiC Whiskers

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Keywords: high-entropy; spark plasma sintering; microstructure; dynamic oxidation.

Abstract

High-entropy carbides (HEC) with varying SiC whiskers (SiC_w) content were synthesized using ball milling and spark plasma sintering (SPS) at 2 100 °C. The study focused on ablation resistance. The ablation behavior of HEC was investigated in this study using an oxyacetylene flame at 2 100 °C. Thermal analysis was performed on the samples to analyze the change in mass during heating, critical temperatures at which possible phase transformations occur, as well as oxidation processes. The results obtained by thermal analysis showed the onset of oxidation for HEC-0SiC_w at ~800 °C, however, with the addition of SiC_w, oxidation starts at a higher temperature of ~860 °C. The results after dynamic oxidation showed that with increasing SiC_w content, the rate of dynamic oxidation decreases (by up to 94.2 % for HEC-10SiC_w compared to HEC-0SiC_w). The addition of SiC whiskers increased the material's resistance to oxidation at elevated temperatures. The surface of the ablated samples was covered with a discontinuous oxide layer, the properties of which varied depending on the SiC_w content (thicker layers of molten metal oxide, a smaller central zone, oxidation products at the center and at the edge). These results suggest that the addition of SiC_w can effectively improve the dynamic ablation resistance of ceramic materials.

Table 1 Surface temperature, oxidation rate of composites during oxidation resistance measurement

	HEC-0SiC_w	HEC-1SiC_w	HEC-3SiC_w	HEC-5SiC_w	HEC-10SiC_w
Weight before oxidation [mg]	6379.4	6742.9	6663.2	6704.9	6676.8
Weight after oxidation [mg]	6478.6	6816.4	6723.5	6753	6669.4
Maximum surface temperature [°C]	2116	2125	2126	2117	2114
Oxidation rate [$mg \cdot cm^{-2} \cdot s^{-1}$]	0.526	0.390	0.320	0.255	0.030

Acknowledgment

The financial support of Slovak Grant Agency VEGA of the Slovak Academy of Sciences (grant no. VEGA 2/0107/24) and of the Slovak Research and Development Agency under the Contracts no. APVV-21-0402 and APVV-22-0493.

Cobalt-Free High-Entropy Oxide $(CrMnFeNiCu)_xO_y$ as a High-Performance Electrode Material for Energy Storage Applications

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Keywords: energy storage; high entropy alloy; high entropy oxide; spinel structure; rock-salt structure; anode.

Abstract

High-entropy materials are among the most discussed topics due to their unique mechanical and functional properties, which arise from the mixture of elements, usually in equimolar composition. Spinel-structured high-entropy oxides are favoured due to their two Wyckoff sites, which increase configurational entropy by accommodating different elements in various oxidation states. On the other hand, rock-salt-structured high-entropy oxides exhibit superior ion diffusion coefficients, enhancing their electrochemical performance. In this work, a dual-phase high-entropy alloy, CrMnFeNiCu, was prepared by arc melting and used as a precursor for synthesizing high-entropy oxide through high-temperature oxidation. The resulting high-entropy oxide exhibited a complex multiphase structure, consisting of two spinel phases and a rock-salt phase, and was investigated as an electrode material for energy storage applications. The HEO-based electrode demonstrated negative fading during cycling, a unique phenomenon observed in advanced conversion-type materials, including high-entropy oxides. A rate capability test revealed the high performance of this oxide material at high current densities, making it suitable for demanding energy storage systems. Post-mortem analysis confirmed the conversion-type mechanism of ion storage and transport during cycling.

Acknowledgment

This work was supported by the Slovak Research and Development Agency under Contract no. APVV-20-0138 and APPVV-MVP-24-0264, and Stefan Schwarz Fund No. 2024/OV1/017. This research was funded in part by the international project M-ERA.NET 3/2022/235/H2MobilHydride.

Development of New High-Entropy Materials for Energy Storage

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Keywords: high-entropy materials; energy storage; anode.

Abstract

The development of advanced materials for energy storage is a fundamental goal in the research of renewable energy sources. High-entropy compounds are distinguished by their high stability, thanks to thermodynamic stabilization, and their structure is affected by fluctuations in composition and the variety of ions with different radii and oxidation states. These materials are synthesized using various techniques that ensure a uniform distribution of elements. Their unique characteristics, such as excellent stability and the potential to optimize both structure and performance, open up new possibilities for energy storage systems. The goal is to extend the durability and efficiency of backup energy sources, which will enable deeper research while maintaining economic and environmental balance. Given these findings, this study focus on the development of a high-entropy material through a more innovative and cost-effective approach. The experimental data shows a positive trend, with increasing storage capacity, indicating that optimized process enhance the material´s qualitative properties.

Acknowledgment

This work was supported by the Slovak Research and Development Agency under Contract no. APVV-23-0030, VEGA project No. 1/0122/25 and KEGA project No. 011TUKE-4/2025. This research was funded in part by the international project M-ERA.NET 3/2022/235/H2MobilHydride.

Microstructural Insights into Medium-Entropy Alloys

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Keywords: hydrogen storage; medium-entropy alloys; metallography; metal hydride; TiZrNbTa-Al.

Abstract

Medium-entropy alloys (MEAs) – multi-principal element systems in near-equimolar ratios (5 - 35 at. %) – provide a versatile foundation for developing hydrogen storage materials. Alloys with a body-centered cubic (BCC) structure are particularly promising materials due to their high number of interstitial sites suitable for hydrogen absorption. This study focuses on the TiZrNbTa system alloyed with Al, aiming to stabilize a single-phase BCC structure in the as-cast condition. Microstructural and mechanical characterization revealed a dendritic, heterogeneous microstructure resulting from arc melting (Fig. 1). XRD analysis indicates the presence of at least two phases, with the primary phase exhibiting a BCC lattice. Density decreased progressively with increasing Al content, while hardness increased up to 7 at. % Al, then slightly declined. These results suggest that homogenization heat treatment is necessary to reduce chemical segregation and enhance structural uniformity, supporting future hydrogen storage evaluation. Future work will focus on hydrogen sorption behavior and evaluating the impact of homogenization on hydrogen storage performance [1].

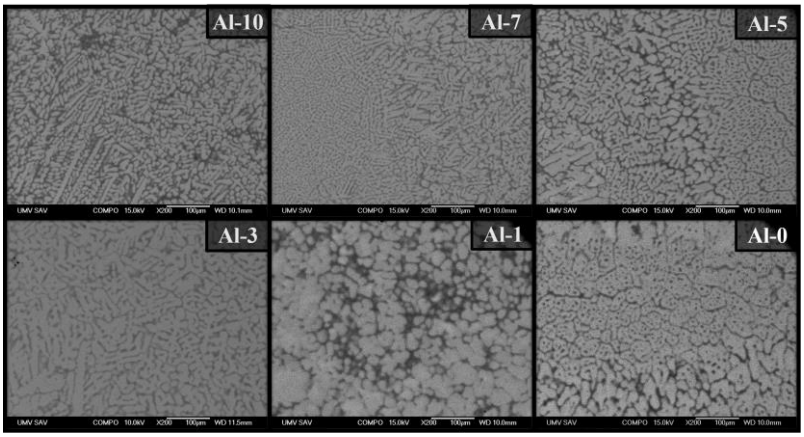

Fig. 1 BSE images showing dendritic, multiphase microstructures of (TiZrNbTa)-Al alloys.

Acknowledgment

This work was supported by the Slovak Research and Development Agency under Contract No. APVV-23-0030, VEGA project No. 1/0122/25 and KEGA project No. 011TUKE-4/2025. This research was funded in part by the international project M-ERA.NET 3/2022/235/H2MobilHydride. The authors also acknowledge support from the project Early Stage Grants TUKE, project code: 09I03-03-V05-00015, under the program "Recovery and Resilience Plan" at the TUKE.

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Study of Carbon Fibers as an Alternative to Graphite in Lithium-Ion Battery Anodes

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Keywords: carbon fibers; graphite; lithium-ion battery; sustainable anode materials.

Abstract

The rapid growth of electric vehicles (EVs) and energy storage systems has intensified the demand for lithium-ion batteries (LIBs) with higher capacity and improved sustainability. Graphite, the conventional anode material, has a limited theoretical capacity (372 $mAh.g^{-1}$) and supply chain challenges, necessitating the development of alternative materials [1]. Carbon-based materials, such as carbon fibers, offer promising electrochemical properties and sustainability benefits.

This study investigates carbon fibers synthesized via low-temperature oxidative catalytic pyrolysis as a potential anode material for LIBs. The synthesis process aligns with the sustainability goals outlined by Batteries Europe, emphasizing the use of inexpensive, renewable raw materials [2] such as cellulose-based material. This approach not only reduces dependence on critical raw materials but also minimizes the environmental impact of battery production.

The synthesized carbon fibers were used as anodes in coin-cell (half-cell) configurations and characterized using electrochemical methods, including galvanostatic charge-discharge cycling, and cyclic voltammetry (CV). Scanning electron microscopy (SEM) was used to observe structural changes before and after cycling. Initial results indicate that carbon fibers achieve an initial capacity of approximately 400–480 $mAh.g^{-1}$, outperforming graphite. Significantly, the individual capacity of the carbon fiber anodes was also rising with higher number of cycles, showing prospects for long-term performance capability with optimization of their morphology and surface chemistry.

These promising results highlight the potential of carbon fibers and similar carbon-based nanomaterials as viable alternatives to traditional graphite anodes in LIBs. With further development and optimization of synthesis methods, particularly through low-temperature oxidative catalytic pyrolysis, these materials could play a crucial role in improving the performance, sustainability, and cost-efficiency of next-generation LIBs.

Acknowledgment

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Recycling of Zinc-Based Industrial Waste into Nanofibrous Material for Potential Wastewater Treatment Applications

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Keywords: recycling waste; electrospinning; zinc oxide; ceramic fibers.

Abstract

Steel production in electric arc furnaces is accompanied by the formation of a fine-grained heterogeneous dust, which is classified as industrial waste. It contains metals such as Fe, Pb, Mn, Ca, Mg, Cu and also significant amount of zinc in the form of ZnO and $ZnFe_2O_4$. The present work describes the preparation and characterization of zinc oxide ceramic fibers with potential for applications in photocatalytic water remediation. The oxide-based fibers were prepared from the precursor solution obtained by the hydrometallurgical treatment of industrial waste product - electric arc furnace dust - by the needle-less electrospinning method and followed by the calcination process. The dust waste was used as input recycling material for the preparation of metal - enriched leachate of $(NH_4)_2CO_3$. Next, an electrospinning solution was prepared based on the given solution using polyvinylpyrrolidone as the carrier polymer, ensuring the formation of the fibrous structure. The as-spun fibers were calcined at 600 °C with 1 hour of dwelling time and air circulation to provide full removal of carbon residues and characterized by XRD, SEM coupled with EDX, and TEM analyses. The resulting porous structure of ZnO fibers contained trace amounts of impurities (Fig. 1a). The fine-grained morphology, consisting of 20-110 nm single-phase ZnO grains (Fig. 1b).

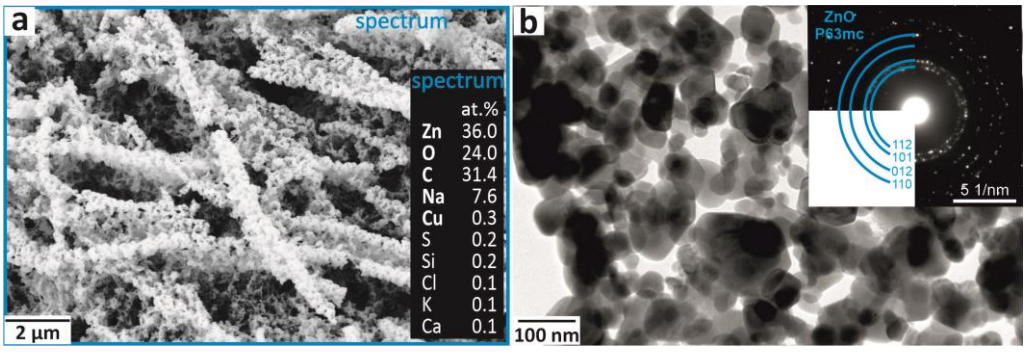

Fig. 1a) SEM image of ZnO fibers with EDX analysis and b) TEM image of ZnO nanograins with pattern of the electron diffraction.

Acknowledgment

This work was supported by the Scientific Grant Agency of the Ministry of Education, Science, Research and Sport of the Slovak Republic and the Slovak Academy of Sciences, project No. VEGA 2/0080/23. The work was funded by the EU NextGenerationEU through the Recovery and Resilience Plan for Slovakia under project No. 09I03 03 V02 00013.

Fracture Surface Morphology and Roughness of Ti-Scaffold Filaments

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Keywords: titanium scaffold; filament; fatigue; fracture morphology; roughness.

Abstract

Scaffolds are advanced 3D metallic porous structures produced by additive manufacturing that find applications in biomedicine as the bone implants [1]. The previous research [2] has shown that the titanium scaffolds with porous filaments (14 % microporosity) exhibited markedly better fatigue resistance than those with compact filaments (5 % microporosity). This was primarily attributed to fatigue crack growth shielding mechanisms [3] and crack path extension induced by interactions between micropores and the advancing crack front.

This article is devoted to macroscopic and microscopic images of fracture surfaces of scaffolds after cyclic compression (CC) tests and porous and of compact filaments after cyclic three-point bending (CTPB) tests using the scanning electron microscopy (SEM) and the confocal optical microscopy (COM). A high density of cracks and broken filaments was particularly indicated in scaffolds with porous filaments. A rather high number of cracking observed also in the scaffolds with compact fibres elucidates a big scatter of CTPB tests of compact fibres as well as a less difference between the S-N curves of compact and porous fibres when compared to the CC tests of scaffolds.

The fatigue crack growth was highly affected by the microporosity in both types of scaffolds. The fracture facets observed by SEM were smaller and rougher for the porous filaments compared to the compact ones. Some cracks followed the pore clusters at grain boundaries of porous fibres to create intergranular facets covered by periodical diffusive patterns. Values of roughness parameters Sa and Sv determined for the fracture surface of the porous filament using COM were significantly higher than those obtained for the compound fibre. A complete statistical roughness analysis will serve as a benchmark for a developed model of fatigue crack growth in porous metals which should enable the optimization of scaffold microporosity with respect to fatigue resistance.

Acknowledgment

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The Influence of Chemical Composition and Plastic Deformations on the Microstructure, Mechanical and Corrosion Properties of the Mg4Zn Alloys

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Keywords: biomaterials; microstructure; mechanical properties; corrosion properties; extrusion.

Abstract

Over the past two decades, significant scientific effort has been devoted to the development and research of bioresorbable metal implants for orthopedic, cardiovascular, and other biomedical applications. These alloys are designed to gradually degrade within the body, eliminating the need for additional surgeries to remove implants, thereby reducing patient discomfort and healthcare costs. Magnesium (Mg) is a biocompatible and biodegradable metal that offers a unique combination of lightweight, strength, and degradation rate that can be tailored to specific clinical needs. However, challenges remain in managing the degradation rate, preventing premature corrosion, and maintaining mechanical stability during the healing process. To overcome these risks, implants made from biodegradable alloys have been developed, which dissolve (in vivo) after completing their function in the host body. The dissolved components have a beneficial effect on the body's healing process, [1]

In this work, we focus on the development of bioresorbable magnesium-based alloys that are alloyed with zinc (4 wt. %) and silver (0.2, 0.4, 0.6, 0.8, and 1.0 wt. %). The advantage of Zn is that it reduces H_2 accumulation and improves the mechanical properties of magnesium biomaterials. Micro-alloying the Mg4Zn alloy with silver enhances its mechanical properties and corrosion resistance, while the antimicrobial properties of Ag also contribute to healing. The positive effect of thermoplastic deformations (extrusion) on the microstructure, mechanical, and corrosion properties of the studied biomaterials has also been confirmed.

Acknowledgment

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Optimizing Mechanical Strength and Corrosion Resistance of Rapidly Solidified Biodegradable Mg-Zn-Ca Alloys: The Ytterbium Addition Impact

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Keywords: biodegradable alloys; magnesium alloys; mechanical properties; met spinning; corrosion resistance.

Abstract

Biodegradable alloys have become a key focus in medical research, with various systems being studied to achieve an optimal balance between mechanical properties and biocompatibility. Magnesium alloys are particularly favored for their low density and excellent mechanical performance. However, their limited corrosion resistance has posed a challenge to broader applications. Recently, magnesium alloys incorporating zinc and iron have gained significant attention due to their appropriate degradation rate.

This study explores the impact of ytterbium (Yb) addition on the mechanical and corrosion properties of the $Mg_{66}Zn_{30}Ca_4$ alloy, which is recognized for its outstanding mechanical strength and biocompatibility. $Mg_{66-x}Zn_{30}Ca_4Yb_x$ (x = 0, 2, 4, 6) rods were produced using a rapid solidification process, and their mechanical and corrosion properties were evaluated against the base alloy. Our findings demonstrate that Yb addition significantly enhances both the mechanical strength and corrosion resistance of Mg-Zn-Ca alloys, making them well-suited for engineering applications requiring high strength and improved corrosion performance. Corrosion analysis revealed that the longitudinal cross-section of the samples exhibited a corrosion depth of approximately 18 μm.

Acknowledgment

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The Microstructure and Mechanical Properties of Mg-Zn Alloy Processed by ECA-Pressing

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Keywords: Mg-Zn alloys; heat treatment; ECA-pressing; microstructure; mechanical properties.

Abstract

Research on biodegradable Mg-Zn alloys has made significant progress in recent years and they are widely used in medical applications [1]. Implants made from these alloys replace conventional permanent metal implants, which have a negative impact on the emotional and physical state of the patients. However, biodegradable Mg-Zn alloys have low strength and plasticity in the as-cast state, and these alloys must be processed using appropriate forming technology before application. One of these techniques is ECA-pressing, a severe plastic deformation technique, which can effectively increase not only the strength but also the plasticity of biodegradable Mg-based alloys [2, 3]. This work presents the results of changes in the microstructure and mechanical properties of the Mg0.1Zn alloy induced by heat treatment and ECA-pressing of the as-cast state of this alloy. The combination of the thermo-mechanical processing of this alloy led to a significant change in the unfavourable coarse-grained solid solution microstructure of the as-cast alloy state and an improvement in its mechanical properties.

Acknowledgment

This research was funded by the Slovak Research and Development Agency under the contract no. APVV-23-0030.

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The Microstructure and Mechanical Properties of Zn Alloy Processed by Severe Plastic Deformation

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Keywords: Zn alloys; microstructure; mechanical properties; ECAP.

Abstract

Zinc-based alloys are considered as promising bioactive materials for bioresorbable orthopaedic implants [1, 2]. Due to their excellent biocompatibility, ability to achieve the required mechanical properties, and a controllable biodegradation, Zn alloys provide an advantageous alternative to conventional metallic materials, such as stainless steel, titanium alloys, and cobalt-chromium alloys, which are commonly used for permanent internal implants. The need to replace these corrosion-resistant materials arises from the "stress shielding" effect, the potential risks of releasing non-biocompatible metal ions, and the requirement for surgical removal of the implant after fulfilling its therapeutic function [3]. This study focuses on a study of the relationship between the microstructure and mechanical properties of the as-cast Zn-Mg alloy state processed by the equal channel angular pressing (ECAP) technique. The obtained results reveal a notable improvement in the mechanical properties of the as-cast Zn-Mg alloy state, which is attributed to the significant modification of its as-cast microstructure induced by severe plastic deformation. The mechanical properties of as-cast alloy state, with an ultimate tensile strength (UTS) about 90 MPa, yield strength (YS) about 80 MPa, and only 2 % elongation to fracture, exhibited a substantial enhancement after ECAP processing. UTS and YS of alloy increased up to 220 MPa and 200 MPa, respectively, and a huge improvement in alloy elongation nearly up to 200 % was found.

Acknowledgment

This research was funded by the Slovak Research and Development Agency under the contract no. APVV-23-0030.

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Application of Industrial Computed Tomography (CT) in Dentistry on 3D Printed Co-Cr Dental Bridge

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Keywords: additive manufacturing; 3D printing; industrial computed tomography (CT); 3D scanning; Co-Cr alloy; dental bridge.

Abstract

Quality control of dental bridges in dentistry is an important area for several key reasons such as maintaining high quality standards, identifying imperfections and volume defects (micro-cracks, porosity and irregularities), checking dimensional accuracy, etc. Industrial computed tomography is of great importance for examination of the above-mentioned characteristics, as it is non-destructive method and the dental bridges do not get damaged or destroyed. In this paper, surface comparison tests were carried out on additively manufactured 3D-printed and casted Co-Cr dental bridges. Precise analysis of the 3D-printed Co-Cr dental bridge internal structure was carried out using industrial computed tomography. Industrial computed tomography analyzed the 3D-printed Co-Cr dental bridge from different angles, such as from the side and in full cross-section. Research has shown various applications and the importance of using industrial computed tomography in the field of quality control of dental bridges. It was determined that it is possible to perform analysis of dimensional geometry (between 3D-printed and cast Co-Cr dental bridge) and the internal structure of the 3D-printed Co-Cr dental bridge. It was found that the 3D-printed Co-Cr dental bridge had no volume defects such as micro-cracks, porosity and irregularities.

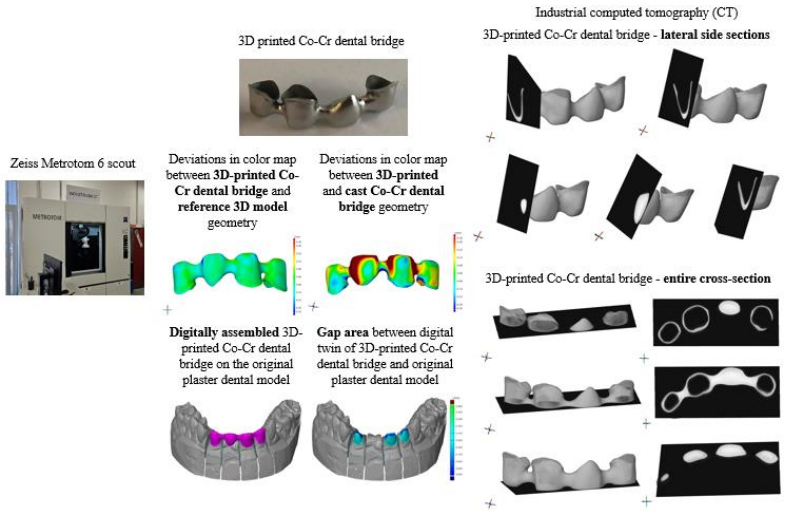

Fig. 1 Industrial computed tomography and 3D scanning in dentistry on Co-Cr dental bridge.

Verification of Concepts for Robotic and Real-Time Etching in Metallography

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Keywords: metallography; etching; automation; robotics; real-time observation.

Abstract

Metallography is a traditional discipline where precision and repeatability in sample preparation are crucial. However, standard etching methods often rely on operator experience, leading to variability in results. This work presents the concept of robotic etching (Fig. 1), which eliminates human influence, ensures consistent timing and movement control during the process, and allows efficient integration with other laboratory procedures. Although robotic etching currently employs predetermined etching times based on prior experience and optimization, significant benefits of this method have already been demonstrated during the optimization stage itself. Particularly for electrolytic etching, this standardized robotic approach consistently achieves reliable and repeatable results, even with materials that are notoriously challenging to prepare. Additionally, real-time monitoring of the sample surface using a stereomicroscope (Fig. 2) (initially tested without robotic integration) offers further potential by enabling termination of the etching process precisely at the optimal moment. Combining robotic control with real-time monitoring thus represents a promising approach with the potential to significantly enhance the repeatability and quality of sample preparation, effectively reducing variability and minimizing the role of chance in the etching procedure. The results validate the feasibility of these methods and highlight their significant potential for enhancing efficiency and quality control in metallography.

Fig. 1 Robotic etching.

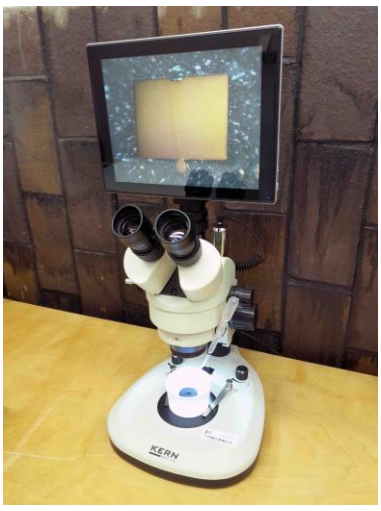

Fig. 2 Etching with stereomicroscope.

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Analysis of Damage Causes of the Steam Heating Element from Tantalum

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Keywords: damage; steam heating element; tantalum.

Abstract

The article deals with determination of damage cause of heating element for material pickling [1-3]. For the analysis parts of heating element were supplied from individual damaged parts. The heating element is immersed in 18 % HCl solution. The heating element is made of tantalum. Heating the HCl solution is provided by hot steam (180 °C) with a working pressure of 10 bar.

Based on analysis results it may be concluded that the primary cause of damage to the heating element of the tantalum is the damage of the flange (Fig. 1a). All other damages are the result of improper repair of this damage (for example Fig. 1b). The flange damage was probably caused when the heating element was installed due to overloading (impact, etc.). Repairing this broken of materials leads to damage whole heating element.

a) b)

Fig. 1 Damaged welding joint of tantalum steam heating element.

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Metallographic Analysis of the Cutting Zone and Comparison with Numerical Simulation

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Keywords: turning; cutting zone; metallography analysis; numerical simulation.

Abstract

Changes in cutting zone during turning are affected by technological conditions of the process. Therefore, it is necessary to know the detailed structural changes in the cutting zone, which can be determined using metallography. Accurate determination of parameters such as shear angle, slip angle, chip thickness, cutting ratio, chip separation point, etc. requires metallographic analysis on a relatively complex sampling of the cutting area. The ideal shape of the cutting zone has a smooth chip, a sharp cutting edge and a sharp transition between the chip and the machined surface as shown in Fig. 1. Based on this, basic parameters such as chip thickness and cutting angle are simply determined for calculating the process characteristics. In real state, the shapes in the cutting zone are more complicated as seen in Fig. 2. The chip thickness must be determined using quantitative metallography from the equality of areas and the resulting point of transition between the chip and the machined surface. The cutting angle starts from this point and is a tangent to the cutting edge, the direction of which must be determined using the Thales circle. The distance between this point and the machined surface represents a layer which is not separated from the machined material but is planar deformed. The depth of the deformed layer and the value of deformation on the machined surface is determined by quantitative metallography. Numerical simulation is simpler, but its results can only be considered valid if they are compared and consistent with the experiment. We performed the analysis on orthogonal cutting. We achieved orthogonal cutting by turning a thin-walled Inconel 718 and C45 alloy tubes and setting the lath bit cutting edge perpendicular to the tube axis. The same parameters were set during the experiment which was metallography analyzed as in the numerical simulation. Only if the numerical mode is verified, numerical simulation can be used instead of metallographic analysis.

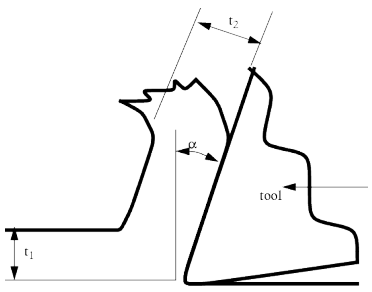

Fig. 1 Cutting zone ideal state.

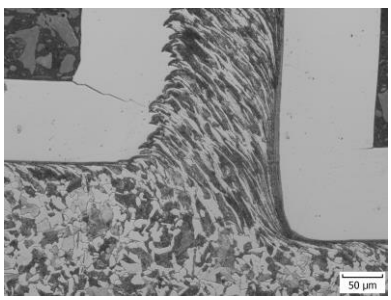

Fig. 2 Cutting zone real state.

Acknowledgment

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Influence of Growth Rate on the Microstructure and Properties of EuBCO Bulk Superconductors Prepared by Top Seeded Melt Growth Process

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Keywords: bulk superconductors; EuBCO; microstructure; properties; growth rate; pores; Eu211 particles; single crystal.

Abstract

To prepare bulk single-crystal REBCO superconductors by the new single-direction growth method (SDMG: Single-Direction Met-Growth), it is necessary to produce a large-area seed of high quality, for example, based on EuBCO. Since the samples prepared by the SDMG method copy the structure of the seed, for the production of large-area seeds it is necessary to optimize the time-temperature regime in order to grow seeds with a suitable structure and composition and minimize structural defects (limiting the amount of subgrains and others).

A higher growth rate was used in comparison with the standard growth rates used to produce EuBCO seeds of larger dimensions. The increased growth rate in the crystal growth window reduces the outflow of the melt from the sample, and thus it is possible to achieve a single-crystal sample in the entire volume of the precursor. The samples were produced at different growth rates: 1; 2; 3 and 5 °C (Fig. 1). The microstructure of the samples was studied by polarized light microscopy and scanning electron microscopy (Fig. 2). The size and distribution of Eu211 particles in the sample volume and the subgrain structure were studied on the fabricated samples.

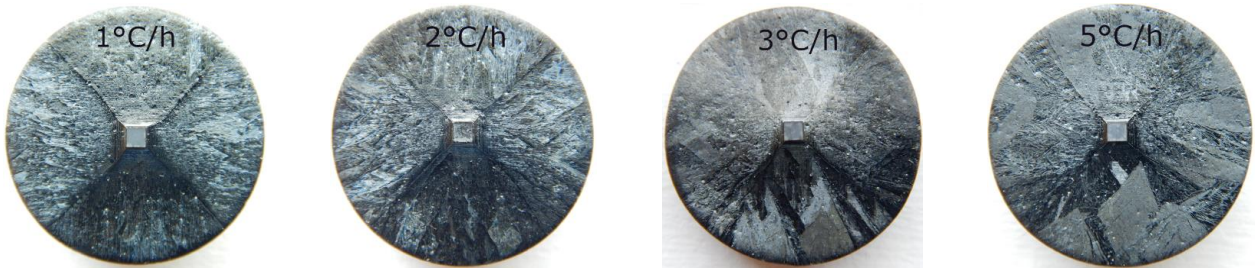

Fig. 1 Macrophotograph of the top of crystals prepared by different growth rates.

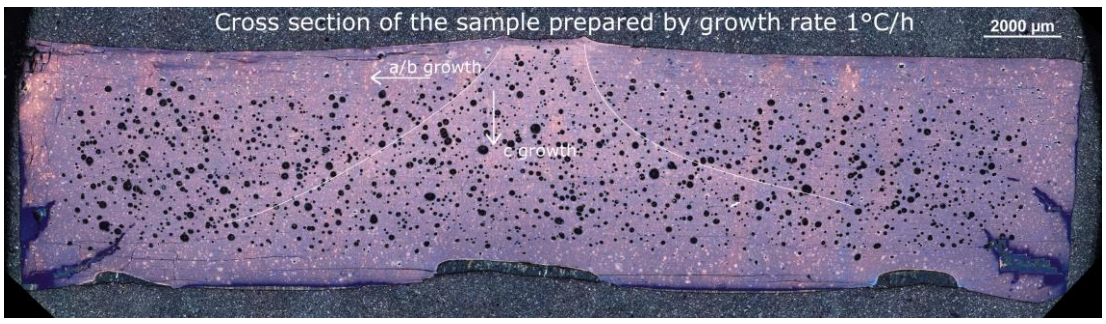

Fig. 2 Cross section of the sample prepared by growth rate 1 °C/h.

Acknowledgment

This work was supported by Slovak Grant Agency APVV-17-0625, APVV-21-0387, No. 2/0094/22 and by NPS/Vojtkova 2025.

Correlation between Additive Composition, Microstructure, and Superconducting Properties in EuBCO Systems

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Keywords: EuBCO-Ag bulk superconductor; microstructure, $BaCeO_3$, BaO_2.

Abstract

This work explores the impact of varying concentrations of $BaCeO_3$ and BaO_2 additives on the microstructure and superconducting properties of bulk EuBCO-Ag materials.

Employing the top-seeded melt-growth technique in air, samples were fabricated. Detailed microstructural characterization, encompassing optical microscopy in polarized light and scanning electron microscopy, was undertaken to investigate the correlation between additive composition and the size, distribution, and density of Eu211 inclusions, Ag particles, and porosity. The influence of these microstructural features on critical current density (Jc), critical temperature (Tc), levitation force, and trapped magnetic field was systematically assessed.

This study seeks to optimize the microstructure and enhance the superconducting performance of EuBCO-Ag bulk materials through a comprehensive understanding of the role of additives.

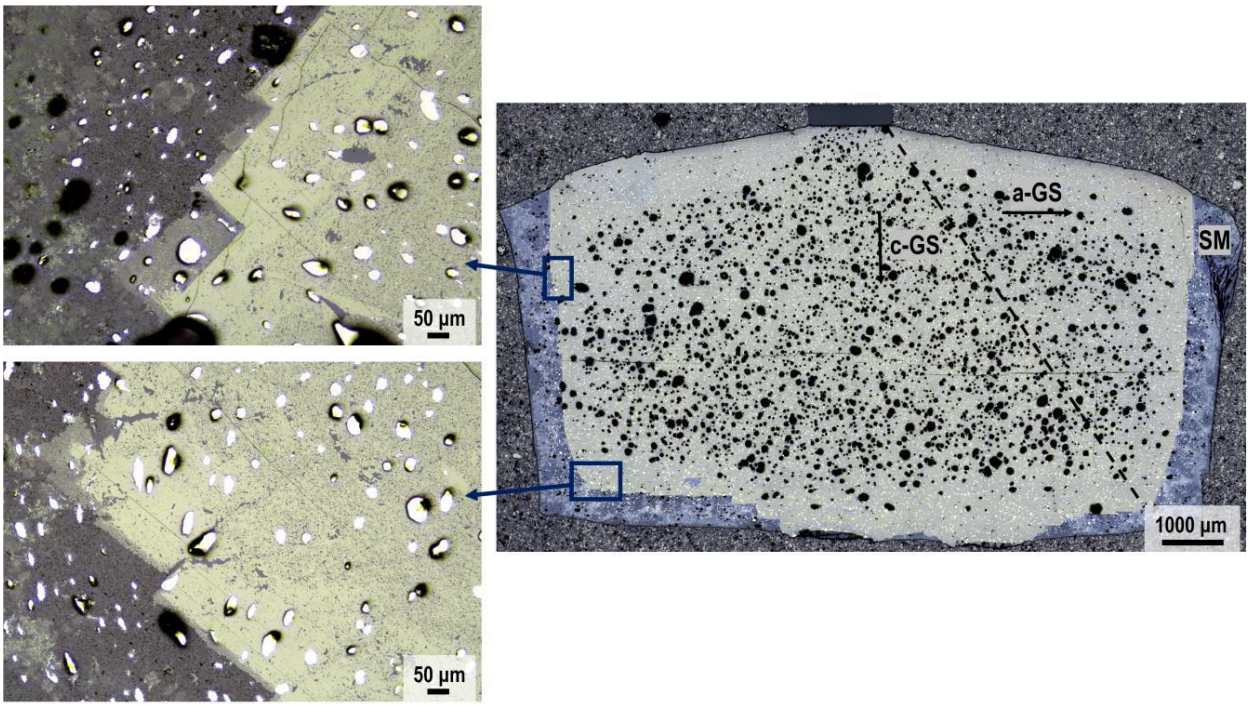

Fig. 1 Polished a/c-sections of sample. Growth sectors (GS) are schematically indicated by dashed line. Solidified melt (SM) at the end of a- and c-GS. Pushing of Eu211 particles has resulted in growth stages in a-GS (top left) and c-GS (bottom left).

Acknowledgment

This work was supported by the European Union NextGenerationEU through the Recovery and Resilience plan for Slovakia under project No. 09I03-03-V04-00303.

Setup and Optimization of a Single Cell PEM Electrolyzer to Produce Green H_2 Using Effective and Cheap Non-Platinum Catalysts

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Keywords: electrolyzer; green hydrogen; non-platinum catalysts; membrane electrode assembly.

Abstract

Research on future proton exchange membrane (PEM) electrolyzers focuses on developing cost-effective, efficient electrocatalysts that match the stability of precious metals for hydrogen evolution reaction (HER) and oxygen evolution reaction (OER) [1]. Non-platinum catalysts, like transition metal phosphides, are gaining attention. Green hydrogen is vital for reducing emissions, storing renewable energy, and replacing fossil fuels in industry due to its high energy density and broad applicability. However, challenges in cost, availability, and efficiency remain, necessitating improved production methods [2]. Water electrolysis is the most common method of producing H_2, with the EU aiming for 14 % renewable energy by 2030. Hydrogen's versatility across industries supports its role as a future fuel, but global production (50 billion m^3/year) is insufficient for rising demand. This research aims to develop and test new electrocatalysts in a single-cell PEM electrolyzer, optimizing into a hydrogen station. Challenges in PEM systems - such as oxidation on current collectors, flow field design, bipolar plate costs, and membrane electrode assembly (MEA) properties - must be investigated to enhance green hydrogen production.

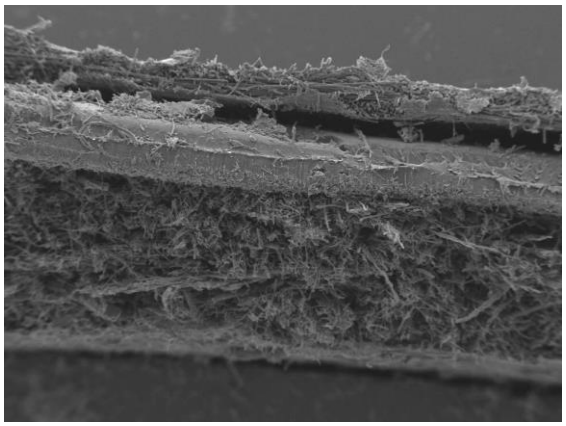

Fig. 1 Cross-section of MEA inside PEM electrolyzer.

Acknowledgment

This work was created with financial support of projects: APVV-20-0299 and VEGA 2/0027/23, VEGA 1/0057/25.

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Effects of Gas Composition and Layer Number on the Mechanical Properties of Multilayered Si-DLC/DLC Coatings

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Keywords: DLC coating; plasma CVD; stainless steel; multilayer structure; surface engineering.

Abstract

In recent years, activities to conserve limited resources have increased, and DLC (diamond-like carbon) coatings, which improve wear resistance and extend the service life of parts, have been attracting attention. DLC is a highly functional film with high hardness and excellent low friction, wear resistance, and corrosion resistance properties; however, it has high residual stress and low adhesion to the substrate. To address these issues, many studies have reported the use of DLC containing metallic elements (Me-DLC) as an intermediate layer to reduce residual stress and improve adhesion. In this study, Si-DLC films were deposited on austenitic stainless steel (SUS304) using the plasma CVD method. Tetramethylsilane (TMS: $Si(CH_3)_4$) and acetylene gas (C_2H_2) were used as source gases for Si-DLC deposition, while acetylene was used for the DLC layers.

Friction and wear tests were performed on samples deposited with Si-DLC and DLC multilayer films with different numbers of layers using the plasma CVD method until the Si-DLC and DLC multilayer films delaminated, without setting a predefined sliding distance, in order to evaluate the durability of the films. The sliding distance before the DLC delaminated is shown in Fig. 1. It is clear that the durability of the film increases with decreasing TMS ratio. As shown in Fig. 2, the decrease in Si and H contents with a lower TMS ratio is considered to increase the hardness and improve the load-bearing capacity of the film. It is also clear that the durability of the films increases with an increasing number of DLC layers (Fig. 1). A possible factor in the improvement of multilayer film properties is the reduction of shear stress at the multilayer interface [1].

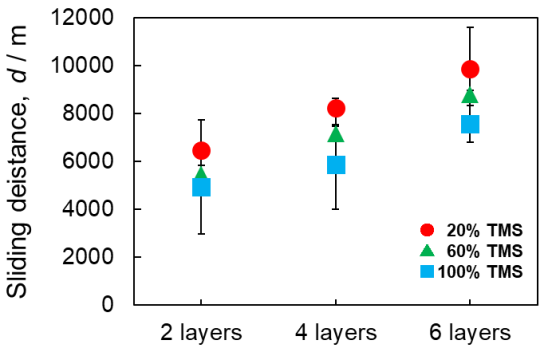

Fig. 1 Delamination distance of various Si-DLC / DLC multilayer.

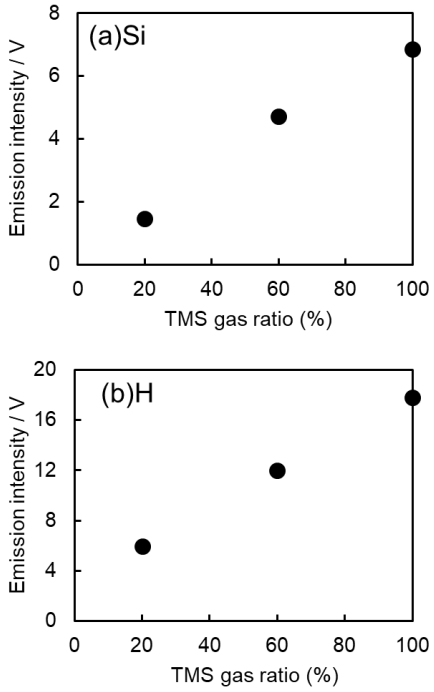

Fig. 2 GD-OES intensity of (a) Si and (b) H as a function of TMS gas ratio.

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Investigation of Al-Si Coating Behavior on 22MnB5 Boron Steel during Heat Treatment

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Keywords: automotive; 22MnB5; Al-Si; heat treatment; intermetallic phases; SEM; EDS; EBSD.

Abstract

22MnB5 high-strength steels are widely used in the automotive industry for manufacturing structural parts of the bodywork. To achieve optimal mechanical properties, these parts undergo quenching, often via hot stamping. An Al-Si based coating is applied to protect the substrate from oxidation during austenitization at high temperatures. This coating undergoes structural changes during the heat treatment, resulting in an inhomogeneous state with varying mechanical properties, which negatively impacts bonding and welding stability.

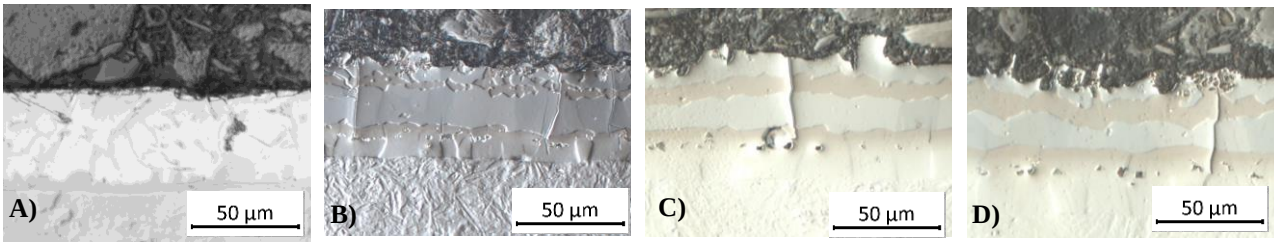

Fig. 1: Structure analysis of Al-Si layer on steel by LOM
A) Default state, B) 920 °C / 420 s, C) 920 °C / 600 s, D) 920 °C / 840 s.

The behavior of the Al-Si coating on 22MnB5 boron steel under different heat treatment conditions was examined. Samples were heated to 920 °C with endurance at the temperature of 420 s, 600 s, and 840 s. Subsequent cooling between two massive iron blocks led to changes in the coating's morphology and the formation of intermetallic phases. Obtained structures were studied using scanning electron microscopy (SEM) equipped with energy-dispersive spectroscopy (EDS) and electron backscatter diffraction (EBSD).

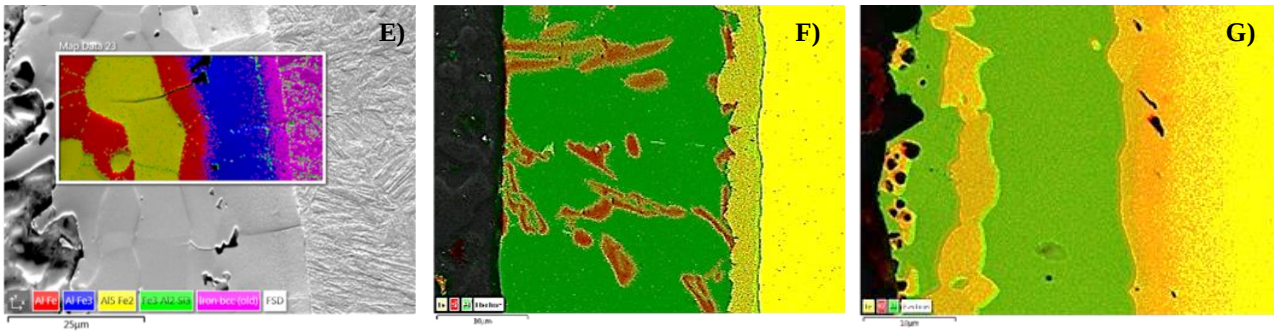

Fig. 2: Structure analysis of Al-Si layer on steel by SEM
E) EBSD map of 920 °C / 840 s, F) EDS map of default state, G) EDS map of 920 °C / 420 s.

These methods proved to be effective for understanding the intermetallic phases formed during manufacturing in Al-Si coatings. The results provide valuable insights into the structural composition and phase transitions of the material, which will guide further research and optimization of these coatings.

Tribological Behavior of ZrO_2-$3Y_2O_3$ Ceramics in Dry Reciprocating Sliding against SiC Counterparts

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Keywords: tribology; Yttria-stabilized Zirconia; wear.

Abstract

The tribological behavior of ZrO_2-$3Y_2O_3$ ceramic sliding against a SiC ball was investigated under dry reciprocating conditions at room temperature. Tests were performed at normal loads of 5 N, 10 N, and 25 N, with a sliding velocity of 0.1 m/s over a total distance of 500 m. The primary aim was to evaluate the coefficient of friction (COF) and wear resistance through detailed wear rate analysis.

The results revealed a distinct run-in phase, followed by a steady-state regime, during which the COF stabilized at 0.39 (5 N), 0.39 (10 N), and 0.37 (25 N). The wear rate exhibited a strong dependence on load, increasing significantly from 9.2783×10^{-8} mm³/N·m at 5 N to 3.1253×10^{-7} mm³/N·m at 10 N, and reaching 5.0539×10^{-7} mm³/N·m at 25 N (Fig. 2). Depth profile measurements confirmed progressively deeper wear tracks (Fig. 1), consistent with elevated mechanical stress levels at higher loads.

Nanoindentation testing revealed a hardness of 16.51 ± 0.86 GPa, indicating that both abrasive and adhesive mechanisms contributed to the overall wear behavior. The transition from mild to severe wear observed at increased loads underscores the critical role of contact pressure in influencing tribological responses.

These findings provide valuable insights into the wear performance of ZrO_2-$3Y_2O_3$ ceramics, highlighting the strong correlation between applied load and wear behavior. This knowledge is essential for the design and optimization of ceramic components intended for dry sliding applications.

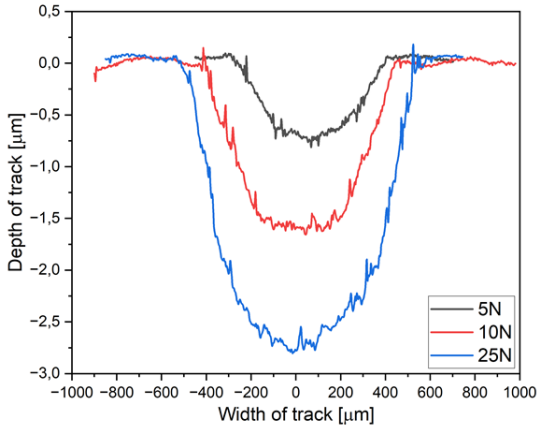

Fig. 1 Characteristic wear profiles of the tested material after the wear test at 5 N, 10 N, and 25 N applied loads.

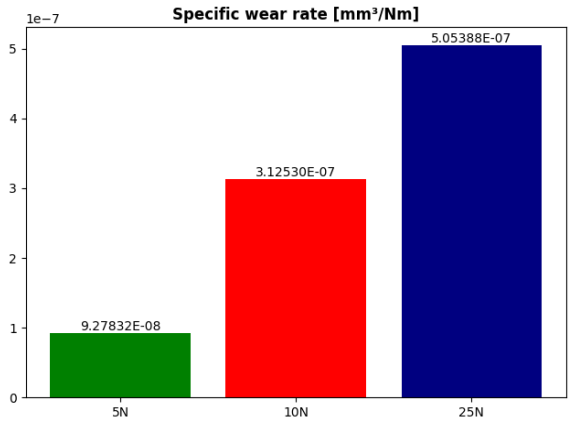

Fig. 2 Wear rates of the investigated ceramic at applied loads of 5 N, 10 N and 25 N.

Acknowledgment

This work was funded by the EU NextGenerationEU through the Recovery and Resilience Plan for Slovakia under the project No. 09I03-03-V04-00260.

Tribological Behavior of Al_2O_3-ZrO_2+3Y_2O_3 Composite

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Keywords: tribology; Al_2O_3; ZrO_2; wear.

Abstract

The tribological performance of a 50:50 Al_2O_3-ZrO_2+3Y_2O_3 composite was evaluated against a SiC ball under dry reciprocating sliding at room temperature. Tests were conducted at normal loads of 5 N, 10 N, and 25 N with a sliding velocity of 0.1 m/s over a 500 m track length to assess the coefficient of friction (COF) and wear resistance.

The COF measurements revealed a higher initial friction during the run-in phase, which stabilized at 0.506 (5 N), 0.483 (10 N), and 0.443 (25 N) in the steady-state phase. Wear profiles showed a progressive increase in wear track depth with increasing load, reaching -1.18 µm (5 N), -1.70 µm (10 N), and -1.97 µm (25 N). The volume of material removed was 1.20×10^6 µm³ (5 N), 2.50×10^6 µm³ (10 N), and 5.30×10^6 µm³ (25 N). Wear rate analysis showed values of

The results indicate a transition from mild adhesive wear at lower loads to increased abrasive wear at higher loads, with the lowest COF and wear rate observed at 25 N, suggesting an improvement in load distribution and potential tribochemical effects. This study highlights the wear behavior of Al_2O_3-ZrO_2+3Y_2O_3 composites, providing valuable insights for their application in high-load dry sliding environments.

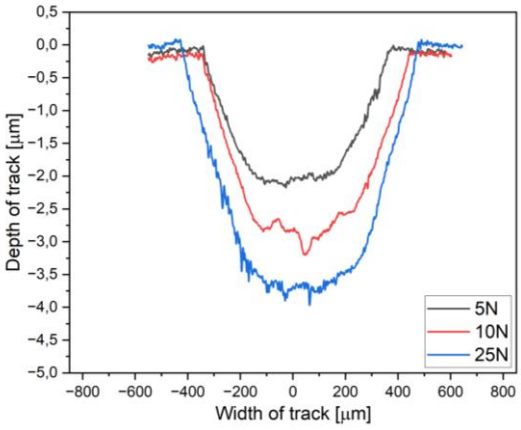

Fig. 1 Comparison of wear profiles for the tested material at 5 N, 10 N, and 25 N loads.

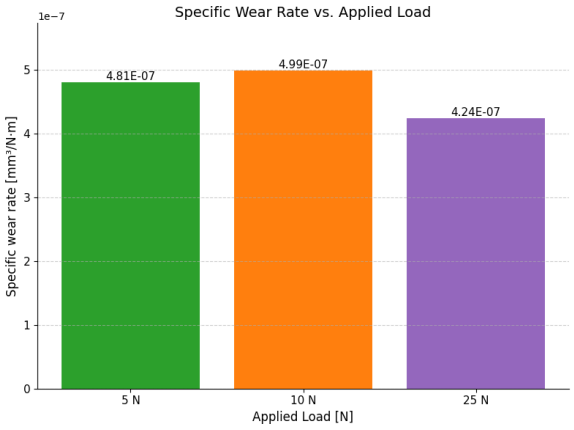

Fig. 2 Comparison of wear rates at applied loads of 5 N, 10 N, and 25 N.

Acknowledgment

This work was funded by the EU NextGenerationEU through the Recovery and Resilience Plan for Slovakia under the project No. 09I03-03-V04-00260.

Gas Pressure Infiltration of Porous Ni-Al_2O_3-Al Compacts with Molten Aluminium

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Keywords: Nickel alumunides; gas pressure infiltration; uniaxial pressing; thermocycling; microstructure.

Abstract

The production of multiphase composite materials based on the Ni-Al system is a complex process that requires careful consideration of several factors, including the strongly exothermic reaction between nickel (Ni) and aluminium (Al). This paper presents a method for fabricating a porous Ni - Al_2O_3 compact using uniaxial doble-action pressing, which was subsequently infiltrated with molten aluminium. Al_2O_3 ceramic particles primarily serve to create porosity within the composite compact. Due to the difficulty in pressing hard metal powders, aluminium powder was introduced into the Ni + Al_2O_3 mixture to act as a plasticizer, improving the material's compressibility. For the compaction process, an optimized experimental powder composition of 84.4 wt. % Ni, 12.5 wt. % Al_2O_3, and 3.1 wt. % Al was selected, with a pressing pressure of 250 MPa. Experiments indicated that the optimal infiltration temperature was 750 °C with an infiltration duration of 300 seconds. To evaluate the reaction extent among the initial components, a subset of infiltrated samples underwent annealing at 800 °C for 3 hours under an inert argon atmosphere. Both annealed and reference samples were subjected to thermal cycling in a dilatometric device, reaching a maximum temperature of 630 °C, again in an inert argon atmosphere. The microstructure of the resulting composite materials was analyzed and characterized using scanning electron microscopy with energy-dispersive spectroscopy (SEM-EDS) (fig.1). Furthermore, the thermal stability of the composites was assessed through repeated thermal cycling.

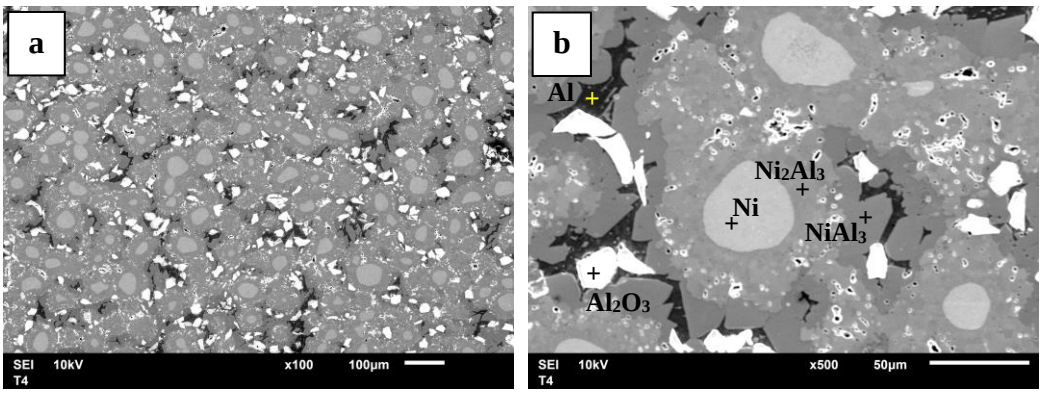

Fig. 1 SEM micrographs of the microstructure of the porous compact after gas pressure infiltration with molten Al at the magnification 100x (a) and detail 500x with EDS point analysis (b).

Acknowledgment

The authors gratefully acknowledge the financial support from the Slovak Grant Agency for Science under the project VEGA No. 2/0061/22.

Deformation Structure of Powdered Iron Compacted at High Pressure

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Keywords: powder compaction; green compacts; microstructure; density; magnetic properties.

Abstract

The pure or insulation-coated iron powders are basic materials for soft magnetic applications. Increasing demands on functional properties, especially high magnetic flux density and as high a cut-off frequency as possible, motivate the development of new compaction technology and the detailed optimisation of traditional uniaxial cold pressing as the most cost-effective solution. Microstructural features as origin particle size and shape, grain size, pores, voids, and lattice defects significantly influence the electromagnetic and mechanical properties of the consolidated soft magnetic materials. High-pressure compaction at pressures higher than 1 GPa can improve green density, increase magnetic filling factor and thus increase permeability. On the other hand, structural defects and residual stresses increase coercivity [1].

The subject of this work is specific deformation microstructure and its distribution in the volume of green compact depending on pressing pressure and shape of the die tool. A metallographically polished upper punch was used for pressing at pressure up to 2.5 GPa to enable direct evaluation of the surface microstructure of green compacts. The influence of the variation of density distribution on functional properties of the soft magnetic materials is discussed.

Figure 1 shows a visualization of the calculated density predicted based on a simple Heckel model respecting the correction for die wall friction.

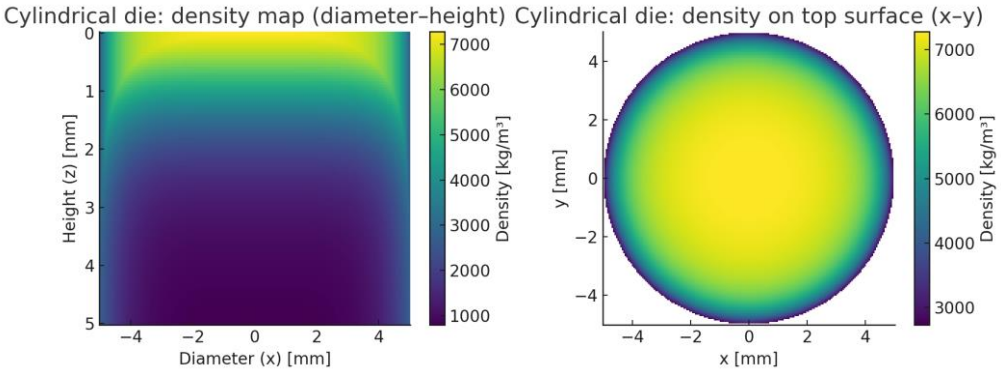

Fig. 1 Calculation prediction heat map of density in cylindrical die.

Acknowledgment

This work was realized within the frame of the projects: APVV-20-0072 and VEGA 2/0099/24.

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Metallography of Sintered H13 Steels Produced by the Additive Method

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Keywords: print 3D; metal FDM; sintering; metallography.

Abstract

Metal Fused Deposition Modelling (Metal FDM) is an emerging incremental technology providing an alternative to selective laser melting (SLM) in the production of metal components. In this study, the microstructure of sintered H13 tool steel produced by Metal FDM was analyses. Two types of filament were used: a commercial Zetamix filament and an experimental filament. Metallographic analysis of the blanks included studies by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) to determine the microstructure and chemical composition of the sinter. The results showed differences in porosity, grain structure and elemental distribution between the materials studied.

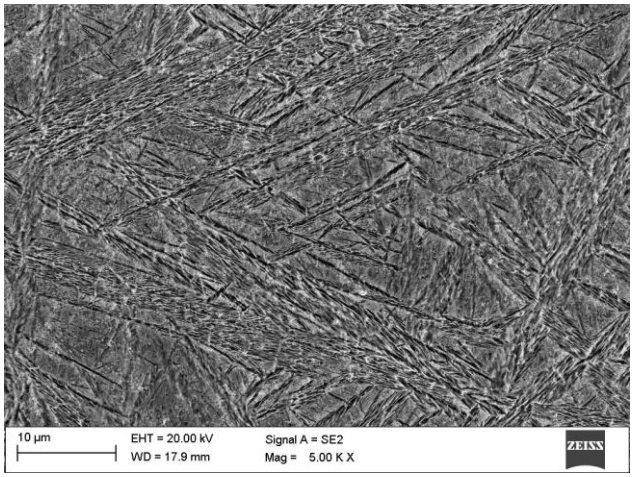

Fig. 1 Microstructure of experimental H13 steel etched in Kalinga reagent, approx. x5k.

Samples of H13 tool steel were etched using Kalling's reagent, which revealed the microstructure of the material. This process allowed for the visualization of grain boundaries and the distribution of individual phases in the steel structure, which allows for the assessment of its homogeneity, grain size and shape, as well as possible microstructural defects.

The analyses conducted indicate the potential of Metal FDM technology in the manufacture of tool steels, while highlighting the microstructural limitations associated with this process.

Acknowledgment

The conference trip was financed by the program of the national academic exchange agency NAWA – PROM.

Comparison of Properties of CeO_2-Doped ZrO_2 Samples Prepared by Conventional Sintering and by SPS

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Keywords: CeO_2-doped ZrO_2; sintering; SPS; shape memory.

Abstract

The aim was to prepare samples suitable for subsequent study of the shape memory phenomenon in the ceramic system ZrO_2 doped with 10 % CeO_2. It was necessary to prepare tetragonally stabilized samples with a grain size of approximately 10 μm. Two methods of sample preparation were chosen, namely conventional sintering and SPS (Spark Plasma Sintering). Conventional sintering was performed at a temperature of 1400 °C (sample E2), or 1600 °C (sample I) for 20 hours. For SPS, the temperature of 2000 °C with holding time 10 minutes was used (sample B). Considering that the sample after SPS was in a cubic modification, subsequent annealing in an oxide atmosphere at 1400 °C for 4 hours was applied (sample B1). This led to the transformation to the tetragonal phase.

The grain size of sample E2 was approximately 1 μm, samples I around 10 μm and samples B and B1 approximately 70 μm (Figure 1).

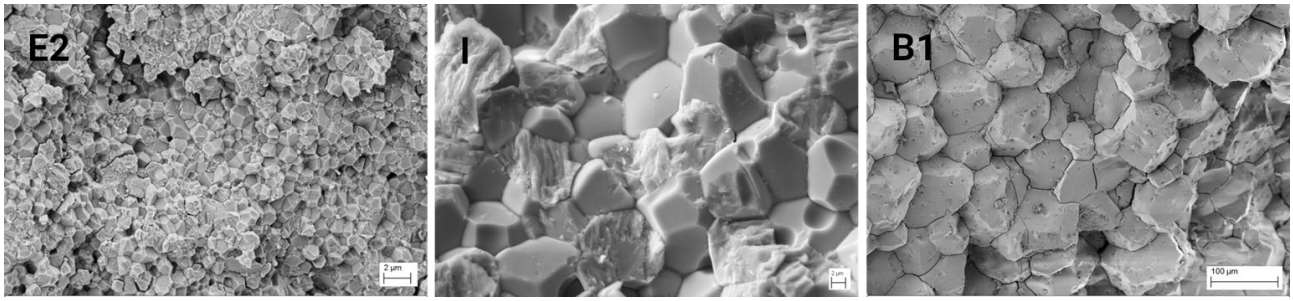

Fig. 1 Fracture surfaces of samples E2 (10000x), I (5000x) and B1 (500x).

The hardness of the samples measured by the nanoindentation and HV methods is given in Table 1. Figure 2 shows the indentations after the HV1 hardness measurement of samples I, around which martensite is visible using polarized light. The fracture strength of sample I was measured on microcantilevers prepared using the SEM/FIB technique. The measured fracture strength of sample I was 2.4 GPa.

Table 1 Hardness of prepared samples

Sample	Hit (GPa)	HV1
E2	1271	938
I	1841	944
B	1877	1168
B1	1237	902

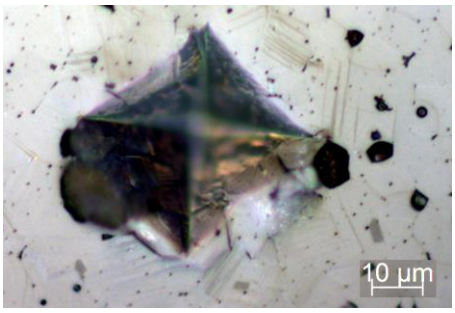

Fig. 2 Sample I, HV1 indent.

Microstructure and Hardness Distribution of Laser Powder Bed Fusion-Produced AISI 2507 Super Duplex Stainless Steel

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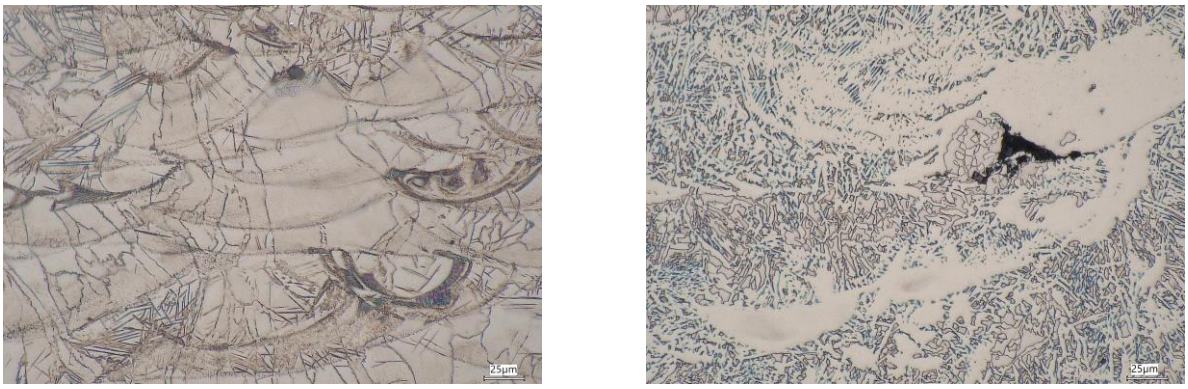
Keywords: SDSS; AISI 2507; LPBF; microstructure; hardness distribution.

Abstract

This study investigated the impact of as-printed and heat-treated additively produced 2507 super duplex stainless steel (also known as SDSS) on microstructure and hardness distribution. Optical microscopy was used to examine the phase transformations of the steel during the as-printed (untreated) and solution-annealed treatment stages of samples. The relationship between microstructure and hardness distribution (core and edge) was studied. Because the LPBF process cools rapidly, the SDSS shows that the main phase in as-printed samples is ferrite, with 5 % austenite. Stress relieving does not affect the phase composition, but it does show a slight increase in the phase of the main austenite. The fully balanced microstructure is forming when the solution annealing is performed, with austenite content about 52 %. The hardness of SDSS is strictly related to the material microstructure, where the fully ferritic structure shows higher hardness 65.2 - 73.2 HRA (41 - 43.2 HRC), while the balanced duplex microstructure reveals lower values 65.0 - 68.1 HRA (23.9 - 29.7 HRC). Measurements at the sample's edge revealed hardness values of 71.9 HRA for as-printed samples and 60.93 HRA for solution-annealed samples.



(a) As-printed (b) Solution annealed

Fig. 1 LOM (light optical microscope) microstructure of SDSS (transverse to build direction XZ).

Table 1 Chemical composition of AISI 2507 super duplex stainless-steel powder [wt. %]

Elements	Fe	Cr	Ni	Mo	Mn	Si	N	Cu	C	P	S	O
[wt. %]	Bal.	24.44	9.11	3.68	0.38	0.29	0.30	0.14	0.01552	0.02328	0.00097	0.01649

Acknowledgment

This work was supported by the Slovak Research and Development Agency under contract No APVV-20-0514.

Effect of Laser Welding on the Microstructural Behavior of Silicon Steels

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Keywords: electrical steels; microstructure; texture; laser welding.

Abstract

Non-oriented FeSi electrical steels are an important class of soft magnetic materials commonly used as core components in various types of rotating electrical equipment. Their soft magnetic properties strongly depend on the ability to control grain size, crystallographic texture, and the chemical composition of the final steel sheets. The most favorable texture for non-oriented silicon steels is the "rotating cube" texture, which ensures isotropic magnetic properties in all in-plane directions of the sheet metal.

Stators and rotors are key components of electric vehicle motors and are composed of hundreds of laminated and joined thin electrical steel sheets. These sheets are designed to be thin (0.2, 0.27, or 0.5 mm) to achieve high magnetic permeability and low iron loss. The laminated electrical steel sheets must be joined at the external circumference to enhance mechanical strength. Both excellent joint strength and optimal magnetic properties are among the most critical performance indicators of the stator. Joining laminated electrical steel sheets can be achieved using three different methods: adhesive bonding, mechanical joining, and fusion welding. Each method has its own advantages and disadvantages, considering its impact on magnetic properties, mechanical properties, and overall cost.

In the present scientific work, the influence of laser welding on the final magnetic properties of joined electrical steels, as well as the evolution of microstructure, thermal stresses, and crystallographic texture around the welding spot, was investigated. Two types of electrical steels with different silicon contents were selected as experimental materials. The experimental samples were prepared in the form of toroids for magnetic measurements after laser welding. The welding was performed along the outer perimeter of five joined toroids, with the samples being coupled using 2, 3, 4, 5, and 6 laser welds.

Acknowledgment

This work was carried out within the research project Funded by the EU Next Generation EU through the Recovery and Resilience Plan for Slovakia under the project No. 09I03-03-V04-00314. Also, the scientific work was supported partly by “Slovak Research and Development Agency” under the contract No. APVV-21-0418 and “Slovak Grant Agency (VEGA)” under the projects VEGA 2/0092/24.

Microstructure Characterization of a Dissimilar 14MoV6-3/P91 Weld after Long-Term Service Exposure at 580 °C

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Keywords: dissimilar ferritic welds; microstructural stability; precipitation; hardness; degradation.

Abstract

Dissimilar ferritic welds are frequently used in modern thermal plants to compensate for local differences in temperature, pressure and corrosive environment. The paper deals with results of microstructure characterization of a dissimilar circumferential weld of 14MoV6-3 and P91 tubes after about 10 years of service exposure in the boiler operated at 580 °C and steam pressure of 10.3 MPa. The P91 tube (ϕ 38 x 4 mm) was welded to the 14MoV6-3 tube (ϕ 38 x 6.3 mm) using the GTAW (141) technology. Bőhler-FOX IN 9-IG (3Cr0.5Mo0.3V) wires were applied as a filler material. The PWHT regime was 740 °C / 1 hour / air.

Microstructural characterization was carried out on longitudinal sections of the dissimilar weld. Investigations were performed using light microscopy, transmission electron microscopy and microhardness evaluation. The very important degradation mechanism of dissimilar welds during their long-term exposure in the creep regime represents redistribution of interstitial elements driven by chemical potential differences of these elements across welds. Table 1 shows results of HV0.5 evaluation in individual parts of the weld. The average microhardness in both carburized areas (WM 3Cr0.5Mo0.3V and CG HAZ P91) did not exceed HV0.05 = 350.

Decarburization of the CG HAZ 14MoV6-3 was accompanied by dissolution of cementite. The high microhardness of this area, compared to the base material not affected by the welding, can be mainly explained by differences in V(C,N) precipitation. The size and interparticle spacing of V(C,N) precipitates was much finer in the CG HAZ 14MoV6-3. Very intensive precipitation was present in both carburized areas. The dominant minor phase in both carburized areas was carbide $M_{23}C_6$. Precipitates in the P91 BM consisted of $M_{23}C_6$, Laves phase and MX, no Z-phase was present.

Table 1 HV0.5 microhardness across the weld

Area	HV0.5 ± STD
BM 14MoV6-3	186 ± 18
CG HAZ 14MoV6-3	252 ± 25
Carburized WM	337 ± 17
WM	301 ± 13
Decarburized WM	253 ± 5
CG HAZ P91	348 ± 10
BM P91	224 ± 11

Results of microstructural characterization of the 14MoV6-3/3Cr0.5Mo0.3V/P91 dissimilar weld after long-term service exposure at 580 °C did not reveal any defects or anomalies in the microstructural evolution during creep exposure.

Acknowledgment

This article was supported by project No.CZ.02.01.01/00/22_008/000463. 'Materials and technologies for sustainable development' within the Jan Amos Komensky Operational Program financed by the European Union and the Czech Republic's state budget.

Evaluation of the Operational Degradation of TVD Pipeline Welds

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Keywords: microbial corrosion; MIC; manganese-oxidizing microorganisms; MOMO.

Abstract

Operational degradation of essential water systems (EWS) pipelines welded joints was evaluated with use of non-destructive radiography and computed tomography (CT), and destructive light optical microscopy (LOM) and scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDS). Large corrosion caverns were confirmed under the inner surface of pipelines. MnO_2 globules found on inner surface near caverns suggest microbial corrosion caused by manganese-oxidizing microorganisms (MOMO) as a potential degradation mechanism.

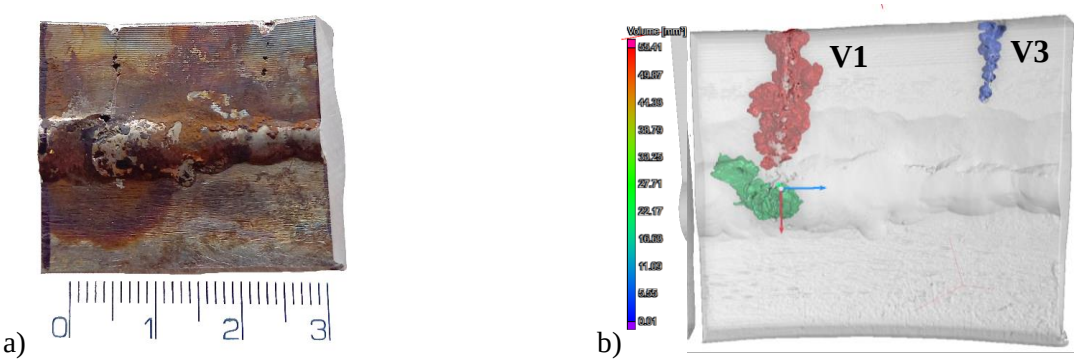

Fig. 1 Inner surface of PS-2 specimen – LOM (a) and its CT scan (b).

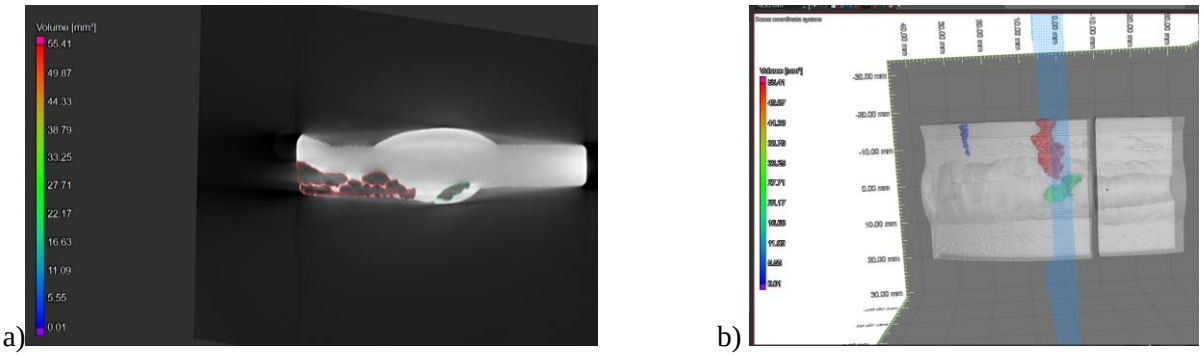

Fig. 2 T cross-section showing the largest extent of V1 (a) CT 3D model with indicated cross-section plane (b).

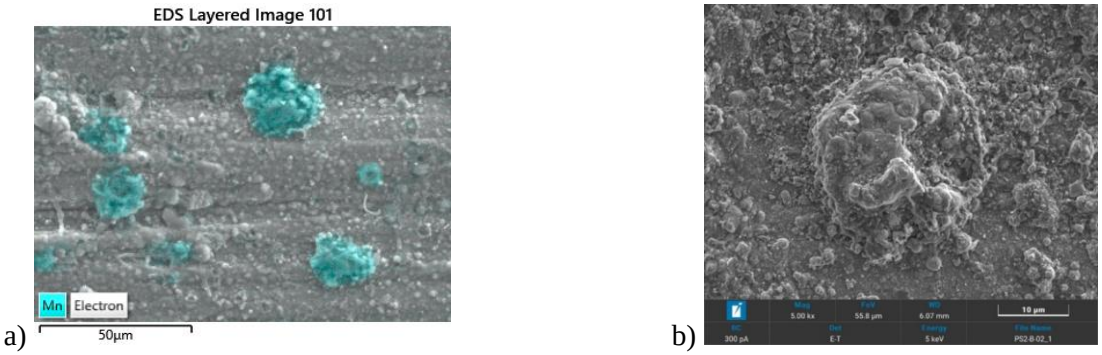

Fig. 3 MnO_2 globules on PS-2-B sample. Manganese composition SEM-EDS map (a) and detail in SEM (E-T detector) (b).

Influence of High-Pressure Hydrogen on Tensile Properties of Pipeline Steels Evaluated by Autoclave In-situ Slow Strain Rate Test

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Keywords: high-pressure hydrogen; SSRT; autoclave; pipeline steel.

Abstract

With search for clean and sustainable energy sources European union is determined to fully substitute natural gas with hydrogen in 2050. This transition should utilize as much of existing gas infrastructure as possible. With these requirements in mind, it is really important to study materials used in existing gas infrastructure and evaluate their resistance to hydrogen embrittlement, which is common phenomena in material degradation when hydrogen is present in some form.

In this article, a comparative study was conducted using slow strain rate testing (SSRT) in situ at 10 MPa hydrogen pressure and tensile testing in air on operated X-52, X-60, and X-70 pipeline steels commonly used in Czech gas infrastructure. Two different strain rates were used for X-60 steel (0.01 and 0.08 $mm \cdot min^{-1}$) to further study strain rate influence in this matter.

The results revealed a decrease in elongation and a significant decrease in contraction in slow strain rate samples tested in hydrogen compared to those tested in air, while yield and tensile strength remained nearly unchanged. These changes are even more prominent with lower deformation speeds. SSRT samples tested in high-pressure hydrogen fracture earlier than those tested in air, meaning less deformation is needed for the fracture itself. Furthermore, a change in fracture morphology occurred. While in air a ductile dimple fracture was observed (Fig. 1), in hydrogen fracture morphology changed to quasi-cleavage/cleavage (Fig. 2). These findings suggest that hydrogen primarily affects the plastic properties of the materials, which ultimately leads to a shift towards a lower energy fracture mechanism.

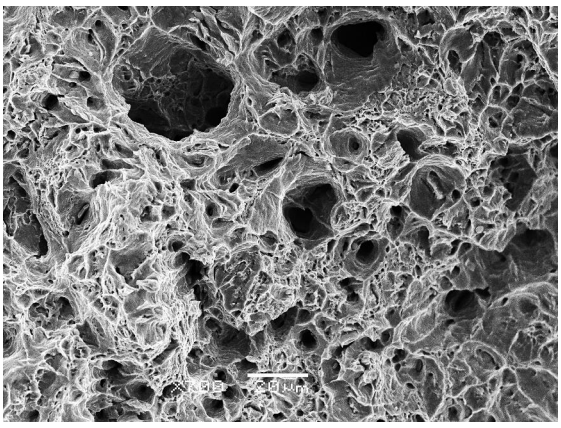

Fig. 1 Micromorphology, X-52, air.

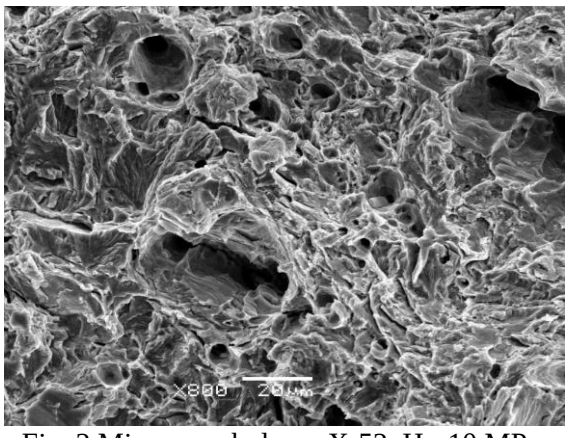

Fig. 2 Micromorphology, X-52, H_2, 10 MPa.

Influence of Thermal Treatments on the Corrosion Behaviour of Nickel-Aluminium Bronze in Freshwater-like Aqueous Environment

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Keywords: nickel-aluminum-bronze; cavitation; etching; freshwater.

Abstract

Nickel-Aluminium Bronze (NAB) is a multi-phased class of copper alloys with excellent mechanical properties and corrosion resistance in marine environments, making it a material of choice for e.g. ship propellers. Consequently, corrosion testing is usually done in seawater-like environments [1]. However, there are also applications of NAB in fresh water and corrosion phenomena have been observed at hydropower turbines [2]. Testing of as-cast NAB in freshwater-typical concentrations of bicarbonate, sulfate, and chloride indicated some susceptibility to various modes of corrosion attack [1, 3].

This manuscript reports corrosion tests with NAB alloy CuAl10Fe5Ni5 thermally treated by annealing 1 hour at 900 °C and water quenched, followed by tempering at 400, 500, or 600 °C for 45 minutes or 24 hours. Potentiostatic corrosion testing at +150 mV_{SCE} was applied for 48 hours in solutions based on combinations of 48 mg/L SO_4^{2-}, 61 mg/L HCO_3^-, 17.5 mg/L Cl^-. Specimens were subjected to metallographic preparation and observation by LOM and SEM.

Results indicate a significant, generally positive impact of the selected thermal treatments on the corrosion behavior which tends to diminish at 600 °C/24 hours. The mayor effect lies in minimization of the martensitic β'-phase amount and increasing the portion of κ-phases. Localized corrosion by sulfate is the major hazard in fresh water, while the passivating effect of bicarbonate supports localization of the attack (Fig.1). Chloride plays an ambivalent role; it induces more general attack but limits the progressive evolution of localized corrosion.

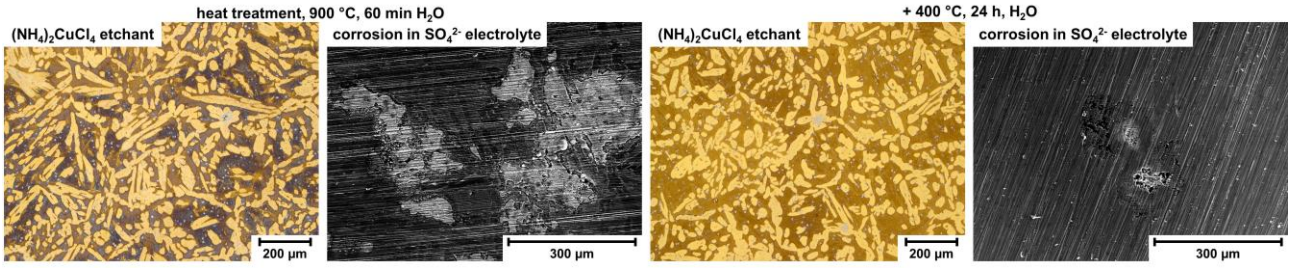

Fig. 1 NAB microstructures after heat treatments (LOM) and surface after corrosion test (SEM).

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Fractography and Microstructural Analysis of Fatigue Crack Propagation in Beta-Annealed Ti6Al4V Alloy after Fatigue Test

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Keywords: Ti6Al4V alloy; beta-annealing; alpha-case layer; fatigue crack initiation and propagation; SEM fractography.

Abstract

Titanium alloy Ti6Al4V was oxidation annealed in the beta-phase region (1050 °C/3 hours + WQ) in a furnace under a non-protective atmosphere. The above treatment caused the formation of an alpha-case layer on the surface. The above layer, because of its high hardness and strength, has a significant effect on the surface properties of the alloy. However, undesirable effects include the formation of cracks in this layer and a change in the mechanism of initiation and propagation of fatigue cracks (as seen in Fig. 1). Based on the above findings, it is also very complicated to predict the fatigue life of Ti alloys processed in this way because of the presence of cracks in the alpha-case layer and the varying thickness of this layer. From the fractography and microstructural point of view, the initiation of fatigue cracks in the heat-treated alloy is realized by transcrystalline cleavage and the formation of pronounced fracture facets as a consequence of surface cracks in the alpha-case layer. Fatigue crack propagation (in the alpha-case layer region) is realized along the interface of the alpha-phase needles and the beta-phase primer grain without the significant presence of so-called fatigue striations.

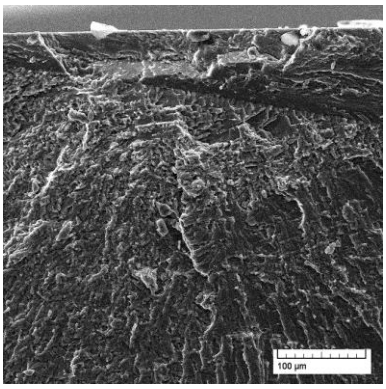
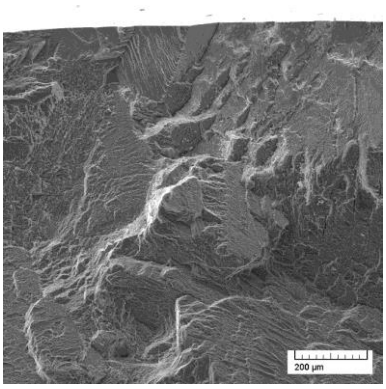
a)  b) 

Fig. 1 Ti6Al4V fatigue crack initiation site at: a) starting stage, b) beta-annealed on 1050 °C/3 hrs. WQ, SEM.

Table 1 The chemical composition (wt. %) and selected mechanical properties of Ti6Al4V alloy (*Ti content is a balance; **Own hardness measurement)

Fe	C	N	H	O	Al	V	UTS [MPa]	Yield strength 0.2% [MPa]	Elongation [%]	Hardness HRC
0.16	0.025	0.009	0.0049	0.164	6.112	4.105				
Long. (not applicable to hardness measurements)							1023	994	12.13	32.5
Trans. (not applicable to hardness measurements)							1185	1152	11.73	57**

Acknowledgement

The authors acknowledge the KEGA projects No. 004ŽU-4/2023 and No. 009ŽU-4/2023 for the financial support of this work.

Fracture Mechanisms of Aluminum Alloy Caused by Fatigue Tests

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Keywords: aluminum alloy; structure; fatigue; fracture; SEM.

Abstract

Material fatigue is one of the main factors affecting the service life and reliability of aluminum alloys in technical applications. This article focuses on the fracture mechanisms of aluminum alloys under cyclic loading, analyzing the initiation and propagation of fatigue cracks. Microstructural aspects such as changes in grain morphology, the presence of intermetallic phases and the influence of casting defects on fracture initiation were investigated. Experimental fatigue tests were performed on a selected aluminum alloy of the Al-Mg type. The results show that the main factors affecting the fatigue behavior are the size and distribution of precipitates, the nature of the interface between phases and the presence of microcracks in the base material. The obtained knowledge provides important information for optimizing the composition and processing of aluminum alloys in order to improve their resistance to fatigue failures.

Acknowledgment

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Metal Dusting Failure in an Aniline Processing Plant

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Keywords: corrosion; carburizing atmosphere; austenitic stainless steel; metal dusting.

Abstract

The paper summarizes the results of the analysis made on the ruptured tube of the reactor made of austenitic stainless steel in which the conversion of aniline to diphenylamine occurs at elevated temperature and pressure and in the presence of a catalyst. The tube wall perforation occurred in the piping used to measure the pressure in the reactor and a fire occurred after an aniline, ammonia and catalyst vapour leak. The material analyses carried out clearly showed that the thinning and subsequent perforation of the tube wall was due to a specific corrosion attack, so-called 'metal dusting', which occurs at elevated temperatures and in the presence of a carburizing atmosphere. The low partial pressure of oxygen in carburizing atmospheres allows the breakdown of the protective oxide layer on the metal surface at elevated temperatures. It happens even in the case of high-alloy steels and nickel alloys which are not immune to this specific type of corrosion attack. In these high-alloy steels, local attack of the metal begins at the point where the protective oxide layer is penetrated, leading to pitting corrosion, while carbon penetrates the material and causes carburization and significant changes of microstructure. The increasing concentration of carbon in the structure was reflected on the metallographic section through the tube wall by firstly highlighting the grain boundaries, secondly by a change in colour to a grey or even black contrast (see Figure 1), but below the tube inner surface the bright contrast gradually returned again due to the precipitation of coarse cementite particles (carbides) along the grain boundaries and within the grains until they eventually reached the character of eutectic carbides, Figure 2. The presence of graphite was then also clearly demonstrated by EDX microanalysis on the inner and highly porous and damaged tube surface.

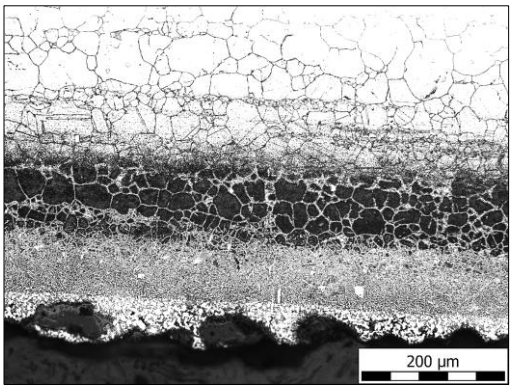

Fig. 1 Increasing carburization towards the tube inner surface.

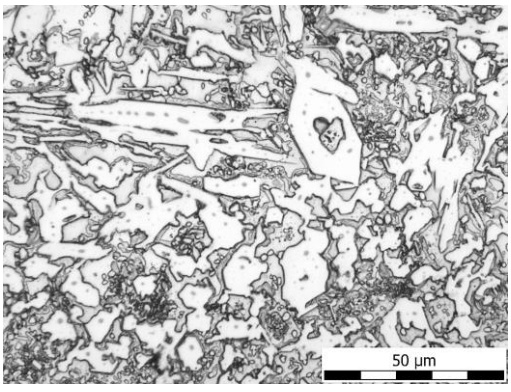

Fig. 2 Detail of the structure with eutectic carbides at the inner surface of the tube.

Acknowledgment

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The Importance of Dislocations in the Work Hardening of Low-Carbon Steel during Cold Plastic Deformation

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Keywords: cold plastic deformation; low carbon steel; digital image correlation; thermography; microstructure; TEM; STEM; dislocation density.

Abstract

It is known that the work hardening process of low-carbon steels is highly dependent on the movement and accumulation of dislocations in the crystal grains, which affect the stress and strain magnitudes and their distribution. The aim of this paper is to explain the importance of dislocation movement and density on the temperature, i.e. stress and strain changes during cold plastic deformation of low-carbon steels. Therefore, tests were carried out in this paper using the methods of static tensile testing, thermography, digital image correlation (DIC) and microstructural analysis. The microstructure analysis was carried out using a light and transmission electron microscope (TEM). The analysis with the transmission electron microscope was conducted in two different operating modes, the TEM and the scanning TEM mode (STEM). The results of static tensile testing, thermography and digital image correlation (DIC) are related to the microstructural changes that occur during the work hardening process of low-carbon steel, Figure 1. At the moment of maximum work hardening (immediately before fracture), significant grain elongation and high dislocation density of low-carbon steel were observed.

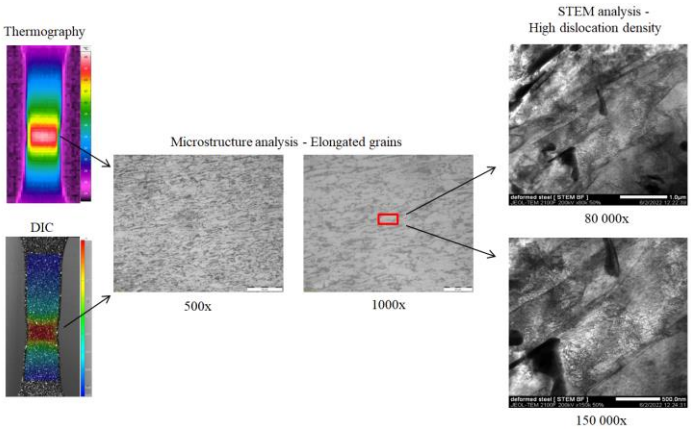

Fig. 1 Maximum work hardening of low carbon steel during cold plastic deformation.

Acknowledgment

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Metallographic and Fractographic Analysis of Plastic Deformation of Austenitic Stainless Steel AISI 304 after Static and Dynamic Loading

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Keywords: austenitic steel; sensitization; microstructure; fracture surface; mechanical loading; plastic deformation.

Abstract

This paper presents a metallographic and fractographic study of 304 austenitic stainless steel subjected to various types of mechanical loading in the sensitised condition. The experimental procedure included three-point bending tests in static and cyclic modes, as well as impact bending tests. The aim was to compare the baseline condition of the material with its condition after sensitisation heat treatment, with particular emphasis on the effects of sensitisation on plastic deformation and fracture mechanisms. Microstructural evaluation was carried out by light microscopy after standard metallographic preparation, while hardness measurements provided complementary information on changes in mechanical response. Fractographic analysis by scanning electron microscopy revealed differences in fracture surface morphology indicating the influence of chromium carbide precipitation on grain boundaries. The results highlight the degradation of mechanical properties and changes in failure modes caused by intergranular sensitisation processes, which are critical for understanding the performance of stainless steels in service conditions involving thermal and mechanical stresses.

Effect of Solution Annealing and Plasma Nitriding on the Fatigue Life Change of AISI 304 Austenitic Steel

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Keywords: austenitic stainless steel; solution annealing; plasma nitriding; microstructure; fractography.

Abstract

Austenitic stainless steels are characterised by excellent corrosion resistance and good formability, but their low hardness and fatigue life are limitations in demanding applications. The aim of this study was to analyze the effect of dissolution annealing and plasma nitriding on the microstructure, hardness and fatigue properties of AISI 304 steel. The experimental material was examined in three states: initial, after dissolution annealing and after plasma nitriding. Dissolution annealing resulted in the removal of deformation martensite, giving a homogeneous austenitic structure with a decrease in hardness. On the contrary, plasma nitriding produced a hard nitride layer (1291 HV0.01), while no martensite retransformation took place. The results of the fatigue tests showed that the specimens after plasma nitriding reached the highest fatigue limit (878 MPa), while the specimens in the initial condition had the highest number of cycles to fracture. Fractographic analysis revealed typical fatigue failure characteristics in all conditions. The study highlights the possibility of optimising the fatigue properties of austenitic steels through an appropriate combination of thermal and chemical-heat treatments.

Failure Analysis of a Copper Pipe Used for Natural Gas Distribution: Investigation of Crack Formation and Fracture Mechanism

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Keywords: fractography; hydrogen embrittlement; Copper piping; brazed joints; EBSD analysis.

Abstract

A defective copper pipe segment used for natural gas distribution was provided for analysis due to gas leakage caused by a crack in the inner radius of a bend (Fig. 1). The investigation focused on identifying the fracture cause, considering material quality, outdated bending technology, and the presence of a brazed joint near the crack. SEM, EDS, and EBSD analyses were employed.

SEM revealed a surface layer partially covering the fracture surface, hindering a full analysis (Fig. 2). Where the layer had detached, no signs of progressive crack propagation were found. EBSD maps (Fig. 3) showed high deformation, dislocation density, and residual stress. The texture corresponds to compression in the bend area.

The crack appeared in close proximity to a brazed joint. EDS analysis indicated the use of L-Ag45 solder (Ag 45 %, Cu 30 %, Zn 25 %), which suggests localized heating to 665 – 745 °C using acetylene. This, combined with the high dislocation density in the heavily deformed material, likely enabled hydrogen ingress. The resulting hydrogen embrittlement triggered intergranular cracking, ultimately leading to failure.

To prevent recurrence, bending radius should be increased, hard brazing avoided near bends, and oxygen-free copper (Cu-OF, Cu-OFE) considered as a more resistant material.

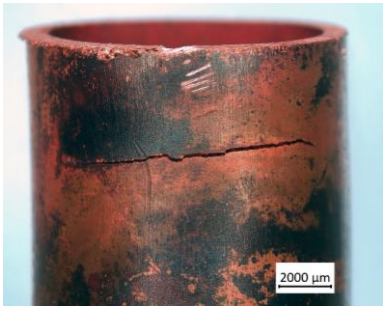

Fig. 1 Provided copper pipe with crack.

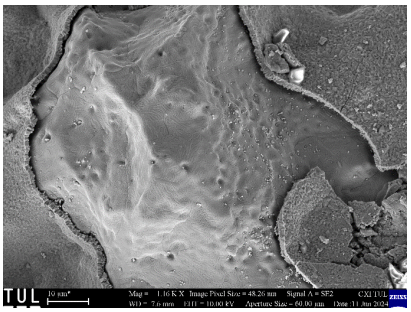

Fig. 2 Fracture surface.

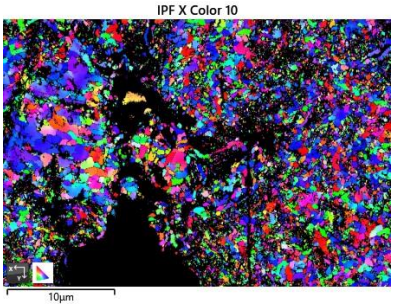

Fig. 3 EBSD analysis of the crack area.

Acknowledgment

This work was supported by the project Support4SME – Grenzüberschreitende Unterstützung für die Beteiligung von KMU an der zukünftigen Materialforschung|Přeshraniční podpora zapojení malých a středních podniků do materiálového výzkumu budoucnosti, project no. 100693265, program „Interreg Sachsen-Tschechien|Česko-Sasko 2021-2027“.

Corrosion Resistance of 3D-Printed AISI 316L Stainless Steel in Molten Glass: Microstructural Evolution and Phase Transformations

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Keywords: 3D printing; additive manufacturing, 316L stainless steel, corrosion resistance, molten glass, high-temperature applications; glass industry.

Abstract

The study investigates the corrosion resistance of 3D-printed AISI 316L stainless steel in LIBA molten glass (produced by Preciosa) at 1032 °C for 96 hours. This study aims to assess the structural stability and phase transformations of 3D-printed stainless steel at the metal-glass interface and in the sample core. The microstructural characterization of samples produced by additive manufacturing was performed before and after the corrosion test in molten glass using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and electron backscatter diffraction (EBSD). The achieved outcomes provide partial information about corrosion resistance, phase stability, and elemental diffusion phenomena at high temperatures. Understanding these mechanisms is crucial for evaluating the applicability of 3D-printed stainless steel components in glass manufacturing, where corrosion resistance and material integrity are essential. The findings contribute to optimising additive manufacturing parameters for high-temperature applications and support the development of durable, corrosion-resistant parts for the glass industry.

Figure 1 below shows a SEM cross-section image of the exposed edge of 3D-printed 316L stainless steel before the corrosion test. Figure 2 also shows the exposed edge, but after exposure to molten glass. Significant structural changes and the formation of new phases at the interface can be observed.

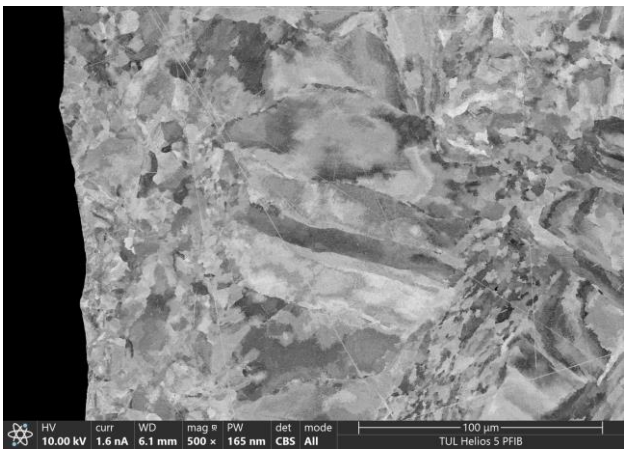

Fig. 1 SEM cross - section image of 3D printed AISI 316L stainless steel - exposed edge AR.

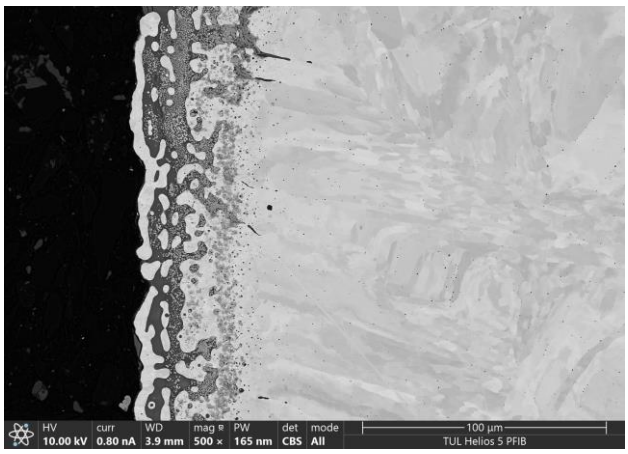

Fig. 2 SEM cross - section image of 3D printed AISI 316L stainless steel - exposed edge after 96 h 1032 °C.

EXHIBITOR PRESENTATION

Instrumentation for Material Research and Quality Control – XRD, SAXS, Particle Size and Shape Analysis, Nanoindentation, Scratch Testing

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Keywords: X-ray diffraction (XRD); small-angle X-ray scattering (SAXS); particle size analysis; scratch testing; nanoindentation.

Abstract

Anton Paar is a globally recognized leader in the field of material research, specializing in the development of high-precision instruments and analytical solutions. The company offers a comprehensive range of advanced technologies for material characterization, including X-ray diffraction (XRD), small-angle X-ray scattering (SAXS), particle size analysis, scratch testing, and nanoindentation. These cutting-edge tools allow researchers to gain in-depth insights into material properties such as structure, composition, mechanical performance, and surface characteristics at both the macro and nanoscale. Anton Paar's solutions are widely used across industries including pharmaceuticals, chemicals, automotive, and semiconductors, providing vital support for quality control, research, and product development. Through continuous innovation and the delivery of state-of-the-art instruments, Anton Paar plays a pivotal role in advancing material science, enhancing research capabilities, and optimizing manufacturing processes worldwide.

Fig. 1 Anton Paar material characterization instruments.

Notes

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ABSTRACT BOOKLET
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