



Microstructure and Properties in Metals and Alloys

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1. Introduction and Scope

Microstructure design is key in targeting the desired material's properties. It is therefore essential to understand the relations between properties and microstructure and how they are driven via a specific process [1–5]. The following five journals contribute to this topic: *Aerospace, Alloys, Crystals, Materials, and Metals*. Contributions related to microstructure design and characterization are collected in this topic, together with their relation to the mechanics, fatigue, wear, and corrosion resistance of different kinds of metals and alloys. The goal of this Topic is to present contributions related to the relationship between the microstructure and properties of metals and alloys for different applications, including aeronautical and aerospace applications. Different process routes are considered (thermo-mechanical routes and additive manufacturing) in this topic. Welding is a mandatory issue in many applications: this is why contributions related to welding are also included in this topic.

2. Contributions

This topic includes 18 articles, two communications and a review paper. Among these, 10 papers were published in *Metals*, 4 papers in *Materials*, 1 in *Alloys*, 3 in *Crystals*, and 3 in *Aerospace*, covering several aspects concerning microstructure properties in metals and alloys.

X. Zhu et al. [6] present a method for the automatic detection of sorbite content in highcarbon steel wire rods. A semantic segmentation model of sorbite based on DeepLabv3+ is established. The sorbite structure is segmented, and the prediction results are analyzed and counted based on the metallographic images of high-carbon steel wire rods marked manually. For the problem of sample imbalance, the loss function of Dice loss + focal loss is used, and the perturbation processing of training data is added. The results show that this method can realize the automatic statistics of sorbite content. The average pixel prediction accuracy is as high as 94.28%, and the average absolute error is only 4.17%. The composite application of the loss function and the enhancement of the data perturbation significantly improve the prediction accuracy and robust performance of the model. In this method, the detection of sorbite content in a single image only takes 10 s, which is 99% faster using the manual cut-off method, which takes 10 min. On the premise of ensuring detection accuracy, the detection efficiency is significantly improved, and the labor intensity is reduced.

Z. Li et al. [7] report in situ observations of the austenite grain growth and martensite transformations in developed NM500 wear-resistant steel conducted via confocal laser scanning high-temperature microscopy. The results indicated that the size of the austenite grains increased with the quenching temperature (37.41 μ m at 860 °C \rightarrow 119.46 μ m at 1160 °C) and austenite grains coarsened at ~3 min at a higher quenching temperature of 1160 °C. Furthermore, a large amount of finely dispersed (Fe, Cr, Mn)3C particles redissolved and broke apart at 1160 °C, resulting in many large and visible carbonitrides. The transformation kinetics of martensite were accelerated at a higher quenching temperature (13 s at 860 °C \rightarrow 2.25 s at 1160 °C). In addition, selective prenucleation dominated, which



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). divided untransformed austenite into several regions and resulted in larger-sized fresh martensite. Martensite can not only nucleate at the parent austenite grain boundaries, but also nucleate in the preformed lath martensite and twins. Moreover, the martensitic laths presented as parallel laths $(0-2^{\circ})$ based on the preformed laths or were distributed in triangles, parallelograms, or hexagons with angles of 60° or 120° .

Z. Wu et al. [8] propose solving the problem of poor metallurgical bonding of Cu/Albimetallic composites caused by high-temperature oxidation of Cu, by different coating thicknesses of Ni layer on Cu rods used to fabricate the Cu/Al bimetallic composite by gravity casting. The effect of liquid-solid volume ratio and coating thickness on microstructure and properties of a Cu/Al bimetallic composite were investigated in this study. The results indicated that the transition zone width increased from 242.3 μ m to 286.3 μ m and shear strength increased from 17.8 MPa to 30.3 MPa with a liquid-solid volume ratio varying from 8.86 to 50. The thickness of the transition zone and shear strength increased with the coating thickness of the Ni layer varying from 1.5 μ m to 3.8 μ m, due to the Ni layer effectively preventing oxidation on the surface of the Cu rod and promoting the metallurgical bonding of the Cu/Al interface. The presence of a residual Ni layer in the cast material hinders the diffusion process of the Cu and Al atom. Therefore, the thickness of the transition zone and shear strength exhibited a decreasing trend as the coating thickness of the Ni layer increased from 3.8 µm to 5.9 µm. Shear fracture observation revealed that the initiation and propagation of shear cracks occurred within the transition zone of the Cu/Al bimetallic composite.

L. Li et al. [9] investigate the impact of various heat treatments on the strength and toughness of TA15 aviation titanium alloys. Five different heat treatment methods were employed in the temperature range of 810–995 °C. The microstructure of the alloy was examined using a scanning electron microscope (SEM) and X-ray diffraction (XRD), and its mechanical properties were analyzed through tensile, hardness, impact, and bending tests. The findings indicate that increasing the annealing temperature results in an increase in the phase boundary and secondary α phase, while the volume fraction of the primary α phase decreases, leading to a rise in hardness and a decrease in elongation. The tensile strength of heat-treated samples at 810 °C was notably improved, displaying high ductility at this annealing temperature. Heat treatment (810 $^{\circ}C/2$ h/WQ) produced the highest tensile properties (ultimate tensile strength, yield strength, and elongation of 987 MPa, 886 MPa, and 17.78%, respectively). Higher heat treatment temperatures were found to enhance hardness but decrease the tensile properties, bending strength, and impact toughness. The triple heat treatment (810 °C/1 h/AC + 810 °C/1 h/AC + 810 °C/1 h/AC) resulted in the highest hardness of 601.3 MPa. These results demonstrate that various heat treatments have a substantial impact on the strength and toughness of forged TA15 titanium alloys.

S. Kusmanov et al. [10] describe how to modify the surface of austenitic stainless steel by anodic plasma electrolytic treatment. Surface treatment was carried out in aqueous electrolytes based on ammonium chloride (10%) with the addition of ammonia (5%) as a source of nitrogen (for nitriding), boric acid (3%) as a source of boron (for boriding), or glycerin (10%) as a carbon source (for carburizing). Morphology, surface roughness, phase composition, and microhardness of the diffusion layers in addition to the tribological properties were studied. The influence of physicochemical processes during the anodic treatment of the features of the formation of the modified surface and its operational properties are shown. The study revealed the smoothing of irregularities and the reduction in surface roughness during anodic plasma electrolytic treatment due to electrochemical dissolution. An increase in the hardness of the nitrided layers to 1450 HV with a thickness of up to 20–25 µm was found due to the formation of iron nitrides and iron-chromium carbides with a 3.7-fold decrease in roughness accompanied by a 2-fold increase in wear resistance. The carburizing of the steel surface leads to a smaller increase in hardness (up to 700 HV) but a greater thickness of the hardened layer (up to 80 μ m) due to the formation of chromium carbides and a solid solution of carbon. The roughness and wear resistance of the carburized surface change are approximately the same values as after nitriding. As a

result of the boriding of the austenitic stainless steel, there is no hardening of the surface, but, at the same time, there is a decrease in roughness and an increase in wear resistance on the surface. It has been established that frictional bonds in the friction process are destroyed after all types of processing as a result of the plastic displacement of the counter body material. The type of wear can be characterized as fatigue wear with boundary friction and plastic contact. The correlation of the friction coefficient with the Kragelsky–Kombalov criterion, a generalized dimensionless criterion of surface roughness, is shown.

Y. Sun et al. [11] study the microstructure and mechanical properties of as-homogenized Mg-xLi-3Al-2Zn-0.2Zr alloys (x = 5, 7, 8, 9, 11 wt.%). As the Li content increased from 5 wt.% to 11 wt.%, the alloy matrix changed from the α -Mg single-phase to α -Mg+ β -Li dual-phase and then to the β -Li single-phase. With the increase in Li content, the alloy strength decreased while the elongation increased, and the corresponding fracture mechanism changed from cleavage fracture to microvoid coalescence fracture. This is mainly attributed to the matrix changing from α -Mg with hcp structure to β -Li with bcc structure. Additionally, the increase in the AlLi softening phase led to the reduction of Al and Zn dissolved in the alloy matrix with increasing Li content, which is one of the reasons for the decrease in alloy strength.

C. Ferro et al. [12] clarify the crucial role of Additive Manufacturing (AM) in the fourth industrial revolution. The design freedom provided by this technology is disrupting limits and rules from the past, enabling engineers to produce new products that are otherwise unfeasible. Recent developments in the field of Selective Laser Melting (SLM) have led to a renewed interest in lattice structures that can be produced non-stochastically in previously unfeasible dimensional scales. One of the primary applications is aerospace engineering where the need for light weights and performance is urgent to reduce the carbon footprint of civil transport around the globe. Of particular concern is fatigue strength. Being able to predict fatigue life in both LCF (Low Cycle Fatigue) and HCF (High Cycle Fatigue) is crucial for a safe and reliable design in aerospace systems and structures. In the present work, an experimental evaluation of compressive–compressive fatigue behavior has been performed to evaluate the fatigue curves of different cells, varying sizes, and relative densities. A Design of Experiment (DOE) approach has been adopted in order to maximize the information extractable in a reliable form.

Y. Guo et al. [13] report on the Nd-Fe-B hot-deformation magnet with high resistivity which was successfully prepared by hot-pressing and hot-deformation of Nd-Fe-B fastquenched powder with amorphous glass fiber. After the process optimization, the resistivity of the magnet was increased from 0.383 m Ω ·cm to 7.2 m Ω ·cm. Therefore, the eddy current loss of magnets can be greatly reduced. The microstructure shows that the granular glass fiber forms a continuous isolation layer during hot deformation. At the same time, the boundary of Nd-Fe-B quick-quenched the flake and glass fiber from the transition layer, which improves the binding of the two, and which can effectively prevent the spalling of the isolation layer. In addition, adding glass fiber improves the orientation of the hot deformation magnet to a certain extent. The novel design concept of insulation materials provides new insights into the development and application of rare earth permanent magnet materials.

G. Stornelli et al. [14] show how the presence of micro-alloying elements in HSLA steels induces the formation of microstructural constituents, capable of improving the mechanical performance of welded joints. Following double welding thermal cycle, with second peak temperature in the range between Ac₁ and Ac₃, the IC GC HAZ undergoes a strong loss of toughness and fatigue resistance, mainly caused by the formation of residual austenite (RA). The present study aims to investigate the behavior of IC GC HAZ of a S355 steel grade, with the addition of different vanadium contents. The influence of vanadium micro-alloying on the microstructural variation, RA fraction formation and precipitation state of samples subjected to thermal cycles experienced during double-pass welding was reported. Double-pass welding thermal cycles were reproduced by heat treatment using a dilatometer at five different maximum temperatures of the secondary peak in the inter-

critical area, from 720 °C to 790 °C. Although after the heat treatment it appears that the addition of V favors the formation of residual austenite, the amount of residual austenite formed is not significant for inducing detrimental effects (from the EBSD analysis the values are always less than 0.6%). Moreover, the precipitation state for the variant with 0.1 wt.% of V (high content) showed the presence of vanadium rich precipitates with size smaller than 60 nm of which, more than 50% are smaller than 15 nm.

S. Najafi et al. [15] study the influence of rare earth (RE) elements on the microstructure and mechanical performance of an extruded ZK60 Mg alloy. Two types of RE elements were added to a ZK60 material and then extruded at a ratio of 18:1. The first new alloy contained 2 wt% Y while the second one was produced using 2 wt% Ce-rich mischmetal. The microstructure, the texture, and the dislocation density in a base ZK60 alloy and two materials with RE additives were studied by scanning electron microscopy, electron backscattered diffraction, and X-ray line profile analysis, respectively. It was found that the addition of RE elements caused a finer grain size, the formation of new precipitates, and changes in the initial fiber texture. As a consequence, Y- and Ce-rich RE elements increased the strength and reduced the ductility. The addition of these two types of RE elements to the ZK60 alloy decreased the work hardening capacity and the hardening exponent mainly due to grain refinement.

C. Xia et al. [16] study coarse particles in Cu-0.39Cr-0.24Zr-0.12Ni-0.027Si alloy with scanning electron microscopy and transmission electron microscopy. Three types of coarse particles were determined: a needle-like Cu5Zr intermetallic phase, a nearly spherical Cr9.1Si0.9 intermetallic phase and (Cu, Cr, Zr, Ni, Si)-rich lath complex particles. The crystallographic orientation relationships of the needle-like and nearly spherical coarse particles were also determined. The reasons for formation and the role of the coarse phases in Cu-Cr-Zr alloys are discussed, and some suggestions are proposed to control the coarse phases in the alloys.

C. Liu et al. [17] study Ta hard coatings prepared on PCrNi1MoA steel substrates by direct current magnetron sputtering, and their growth and phase evolution could be controlled by adjusting the substrate temperature (T_{sub}) and sputtering power (P_{spu}) at various conditions ($T_{sub} = 200-400$ °C, $P_{spu} = 100-175$ W). The combined effect of T_{sub} and P_{spu} on the crystalline phase, surface morphology, and mechanical properties of the coatings was investigated. It was found that higher P_{spu} was required in order to obtain α -Ta coatings when the coatings are deposited at lower T_{sub} , and vice versa, because the deposition energy (controlled by T_{sub} and P_{spu} simultaneously) within a certain range was necessary. At the optimum T_{sub} with the corresponding P_{spu} of 200 °C-175 W, 300 °C-150 W, and 400 °C-100 W, respectively, the single-phased and homogeneous α -Ta coatings were obtained. Moreover, the α -Ta coating deposited at T_{sub} - P_{spu} of 400 °C-100 W showed a denser surface and a finer grain, and as a result exhibited higher hardness (9 GPa), better toughness, and larger adhesion (18.46 N).

M. Cohen et al. [18] analyze an assemblage of tiny provincial silver coins of the local (Judahite standard) and (Attic) obol-based denominations from the Persian and Hellenistic period Yehud and dated to the second half of the fourth century BCE to determine their material composition. Of the 50 silver coins, 32 are defined as Type 5 (Athena/Owl) of the Persian period Yehud series (ca. 350–333 BCE); 9 are Type 16 (Persian king wearing a jagged crown/Falcon in flight) (ca. 350–333); 3 are Type 24 series (Portrait/Falcon) of the Macedonian period (ca. 333–306 BCE); and 6 are Type 31 (Portrait/Falcon) (ca. 306–302/1 BCE). The coins underwent visual testing, multi-focal light microscope observation, XRF analysis, and SEM-EDS analysis. The metallurgical findings revealed that all the coins from the Type 5, 16, 24, and 31 series are made of high-purity silver with a small percentage of copper. Based on these results, it is suggested that each series was manufactured using a controlled composition of silver–copper alloy. The findings present novel information about the material culture of the southern Levant during the Late Persian period and Macedonian period, as expressed through the production and use of these silver coins.

A. Khajesarvi et al. [19] study the effects of carbon, Si, Cr, and Mn partitioning on ferrite hardening using a medium Si low alloy grade of 35CHGSA steel under ferrite-martensite/ferrite-pearlite dual-phase (DP) conditions. The experimental results illustrated that an abnormal trend of ferrite hardening had occurred with the progress of ferrite formation. At first, the ferrite microhardness decreased with increasing volume fraction of ferrite, thereby reaching the minimum value for a moderate ferrite formation, and then it surprisingly increased with subsequent increase in ferrite volume fraction. Beside a considerable influence of martensitic phase transformation induced residual compressive stresses within ferrite, these results were further rationalized in respect of the extent of carbon, Si, Cr and Mn partitioning between ferrite and prior austenite (martensite) microphases leading to the solid solution hardening effects of these elements on ferrite.

Q. Liu et al. [20] investigate the anchoring performance of a short-lapped-rebar splice with a corrugated metal duct and spiral hoops. A total of 30 specimens were designed considering the influences of the rebar diameter and the lapped length, and the tension testing of the splice was carried out. The results show that the specimens with 0.15 times the suggested length in GB 50010-2010 fail by the fracture of rebar, while the specimens with 0.1 times and 0.05 times the suggested length show the pull-out failure of rebar. The ultimate bond strength of specimens with the suggested length is higher than that of the conventional specimens. The stress of the anchored rebar in the short-lapped-rebar splices is distributed symmetrically along the longitudinal direction. The maximum bond stress of the anchored rebar reaches 35 MPa, which is approximately 1.4 times higher than in the conventional specimens. A semi-empirical model for predicting the ultimate bond strength of the short-lapped-rebar splice is proposed, and it shows good agreement with tested values; the average error estimated from the proposed model is only 4.49%.

R. Canumalla et al. [21] evaluated, ranked, and selected near- α Ti alloys from the literature for high-temperature applications in aeroengines driven by decision science by integrating multiple attribute decision making (MADM) and principal component analysis (PCA). A combination of 12 MADM methods ranked a list of 105 alloy variants based on the thermomechanical processing (TMP) conditions of 19 distinct near- α Ti alloys. PCA consolidated the ranks from various MADMs and identified top-ranked alloys for the intended applications as: Ti-6.7Al-1.9Sn-3.9Zr-4.6Mo-0.96W-0.23Si, Ti-4.8Al-2.2Sn-4.1Zr-2Mo-1.1Ge, Ti-6.6Al-1.75Sn-4.12Zr-1.91Mo-0.32W-0.1Si, Ti-4.9Al-2.3Sn-4.1Zr-2Mo-0.1Si-0.8Ge, Ti-4.8Al-2.3Sn-4.2Zr-2Mo, Ti-6.5Al-3Sn-4Hf-0.2Nb-0.4Mo-0.4Si-0.1B, Ti-5.8Al-4Sn-3.5Zr-0.7Mo-0.35Si-0.7Nb-0.06C, and Ti-6Al-3.5Sn-4.5Zr-2.0Ta-0.7Nb-0.5Mo-0.4Si. The alloys have the following metallurgical characteristics: bimodal matrix, aluminum equivalent preferably ~8, and nanocrystalline precipitates of Ti3Al, germanides, or silicides. The analyses, driven by decision science, make metallurgical sense and provide guidelines for developing next-generation commercial near- α Ti alloys. The investigation not only suggests potential replacement or substitute for existing alloys but also provides directions for improvement and development of titanium alloys over the current ones to push out some of the heavier alloys and thus help reduce the engine's weight to gain advantage.

Q. Zhao et al. [22] study the effect of the trace rare-earth element Ce on the microstructure and properties of cold-rolled medium manganese steel after ART (austenite-reverted transformation, ART) annealing was studied. The microstructure of the experimental steel was observed using SEM, and the mechanical properties were tested using a universal tensile testing machine. The volume fraction of the retained austenite and the texture of the steel were measured using XRD. The results showed that the original austenite grain size of the experimental steel was smaller after adding the trace rare-earth element Ce. After ART annealing, the grain size distribution of the experimental steel with rare-earth Ce was more uniform, and the comprehensive mechanical properties were better. Under the conditions of quenching at 800 °C for 5 min and annealing at 645 °C for 15 min, the maximum product of tensile strength and elongation was 28.47 GPa%.

W. Wang et al. [23] report on the dendritic growth and physical properties of broad-temperature-range Co-4.54%Sn alloy. The maximum undercooling attains 208 K at molten

state, and the dendritic growth velocity is quite sluggish in highly undercooled liquid Co-4.54%Sn alloy because it has a broad solidification range of 375 K (0.21 TL); the maximum value is only 0.95 m/s at the undercooling of 175 K, which then decreases with undercooling. The microstructure refines visibly and the volume fraction of the interdendritic β Co3Sn2 phase clearly decreases with undercooling. The microhardness and electrical resistivity increase with undercooling owing to the enhancement of solute content of the primary α Co phase and refinement of the microstructure where the increased crystal boundary hinders the electronic transmission. Meanwhile, the saturation magnetization also reduces with undercooling due to the crystal particle and boundary increasing significantly, and the dendritic growth velocity and solute content increase in the primary α Co phase under rapid solidification.

T. Tretyakova et al. [24] study the influence of the rigidity of the loading system on the kinetics of the initiation and propagation of the Portevin-Le Chatelier (PLC) strain bands due to the jerky flow in the Al-Mg alloy. To estimate the influence of the loading system, the original loading attachment, which allows for a reduction in the stiffness in a given range, was used. Registration of displacement and strain fields on the specimen surface was carried out via the Vic-3D non-contacting deformation measurement system based on the Digital Image Correlation (DIC) technique. The mechanical uniaxial tension tests were carried out using samples of Al-Mg alloy at the biaxial servo-hydraulic testing system Instron 8850. As a result of tensile tests, deformation diagrams were obtained for Al-Mg alloy samples tested at different values of stiffness of the loading system: 120 MN/m (nominal value), 50 MN/m, 18 MN/m, and 5 MN/m. All diagrams show discontinuous plastic deformations (the Portevin-Le Chatelier effect). It is noted that a decrease in the rigidity of the loading system leads to a change in the type of jerky flow. At constant parameters of the loading rate, temperature, and chemical composition of the material, the PLC effects of types A, B, and C are recorded in tests.

T. Hu et al. [25] describe the diffusion of TM and the nucleation and growth of particles in Al alloys based on first principles.

M. Gaggiotti et al. [26] present a review on the effect of ultrafast heating (UFH) treatment on carbon steels, non-oriented grain (NGO) electrical steels, and ferritic or austenitic stainless steels. The study highlights the effect of ultrarapid annealing on microstructure and textural evolution in relation to microstructural constituents, recrystallization temperatures, and its effect on mechanical properties. A strong influence of the UFH process was reported on grain size, promoting a refinement in terms of both prior austenite and ferrite grain size. Such an effect is more evident in medium-low carbon and NGO steels than that in ferritic/austenitic stainless steels. A comparison between conventional and ultrafast annealing on stainless steels shows a slight effect on the microstructure. On the other hand, an evident increase in uniform elongation was reported due to UFH. Textural evolution analysis shows the effect of UFH on the occurrence of the Goss component (which promotes magnetic properties), and the opposite with the recrystallization g-fiber. The recovery step during annealing plays an important role in determining textural features; the areas of higher energy content are the most suitable for the nucleation of the Goss component. As expected, the slow annealing process promoted equiaxed grains, whereas rapid heating promoted microstructures with elongated grains as a result of the cold deformation. It is worth mentioning, and may be useful for readers, that, after this review, the same authors published experimental investigations related to the effect of ultra-fast heating on stainless steels [27,28].

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