

How nitrogen alloying affects the structure of multilayer metal materials

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Abstract. This article deals with the way nitriding affects the structure of multilayer metal materials. The subject of research was a composition of alternating layers of the U8 carbon steel and austenitic 08H18n10 grade steel with various structuring degrees. Key words: chemical and thermal processing, nitriding, grain boundary diffusion.

1 Introduction

Multilayer metal materials represent point of significant interest for conducting diffusion processes due to the presence of laminar structure and the ability to achieve various degrees of its structuring in the course of a technological cycle.

Multi-material hybrid structures in nature and technology combine properties of their individual constituents throughout complex microstructures to achieve synergistic effects, like improved mechanical properties and oxidation resistance, which go beyond the properties of their monophasic counterparts. Typically, multilayered, composite and hierarchical microstructures are used to achieve improved functionality by combining materials with different physical properties. Similarly, functionally graded materials (FGMs) are designed to comply with spatially variable functional requirements like strength, oxidation resistance or toughness by adopting gradients of dissimilar phases, microstructure and/or residual stresses. However, joining of metals into multimetal hybrid structures with diffuse or abrupt interfaces between the individual phases or alloys and narrow heat affected zones resulting in limited the first-order residual stress, macroscopic distortion and secondary phase formation represents still a serious technological challenge. [1]

By their principle of a sequential layer-by-layer deposition, additive manufacturing technologies (AM) are ideally suited to manufacture parts with complex external and internal geometries. Additionally, powder and wire feed material supplies have been known to allow for an adjustment of volume fraction of metallic components. In particular, the directed energy deposition (DED) approach, in which powder is fed into the melt-zone and molten at every layer by a laser, can be used to fabricate components with a variable layer-by-layer composition and unique microstructures. Within this contribution, however, a novel laser

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powder bed fusion-related technology, based on liquid dispersed metal powder, is used to produce a multi-metal hybrid structure. [1]

The fabrication of FGMs using AM technologies allows for generating property-specific part areas by matching the process parameters to the localized functionality. It is however common that combining dissimilar metals using AM technology may result in the formation of compositional and microstructural inhomogeneities as well as in secondary phases and stress concentrations, which may decisively deteriorate part's functional properties. [1]

A significant effort has been devoted to the development of multi-material additive manufacturing (MM-AM) of hybrid structures. Exemplary AM systems equipped with several powder feeders were used to prepare graded Ti-V, Ti-Mo, V-Ti6Al4V alloy, stainless steel-Ti6Al4V alloy and Ti-6Al-4 V/Ti-6.5Al-3.5Mo-1.5Zr-0.3Si materials with novel refined microstructural features, aiming at aircraft and aero-engine applications. Similarly, bimetallic structures like Inconel 718-copper alloy GRCop-84 and stainless steel 304 L-Invar 36 as well as gradient structures like Ti-TiO₂ and yttria-stabilized zirconia-steel were fabricated. [1]

In the field of nuclear and steam power plants, aerospace and/or repair applications, especially high strength, corrosion and oxidation resistance and/or creep and fatigue resistance are required. For this reason, MM-AM of stainless steel (SS) and Inconel have been extensively explored. Carroll et al. [1] reported on fabrication of Inconel-steel FGM with diffuse compositional, structural and microstructural boundaries by applying gradually varying mixtures of Inconel 625 and grade 304L SS powders. The observed formation of cracks with a length of several hundred microns in a region with 79 wt% SS 304L and 21 wt% IN625 was attributed to the presence of secondary phases of transition metal carbides in the form of (Mo,Nb)C. Similarly, aiming at nuclear fission reactor applications, Hinojos et al. [36] studied the joinability of Inconel 718 and 316L SS and vice versa utilizing electron beam melting (EBM) and reported abrupt compositional, structural and microstructural boundaries as well as low concentration of typical welding features. Cracking observed at the 316L/IN718 interface was attributed to the deformation constrains imposed by IN718. Additionally, using finite-element numerical analysis, Hofmann et al. [1] showed that a gradient transition from SS 304L to IN625 across an automobile valve stem are expected to exhibit ten times lower stress at the transitioning zone at 1000 K compared to a friction stir-welded joint of the same materials. [1]

In general, all previous studies on FGMs combining nickel-based alloys and steel indicated complex process-microstructure-stress-properties relationships. Additionally, FGMs produced with diffuse and abrupt interfaces comprise usually unique microstructures with secondary phases and precipitates like (Mo,Nb)C, M₂₃C₆, NbC etc. and there were cracks with lengths up to several 100 μm observed systematically in the transition regions. These findings indicate that there is a significant potential to further optimize the functional properties of the particular nickel-based alloys-steel FGMs by knowledge-based microstructural design, including a development of novel deposition routes and recipes. [1]

As it is known, the progress of various reactions related to the microstructure changes occurs through diffusion processes, whereas the speed of diffusion is determined by the presence of imperfections in the crystal structure and structural defects. The principal elements ensuring the speed of the diffusion are the grain boundaries [2-3]. The grain boundaries play a significant part both in the recrystallization processes and in the chemical and thermal processing.

2 Research methods and materials

There exist various methods of producing multilayer materials [4-6], however, this project deals with the multilayer metal materials produced through hot package rolling, which results in the laminar structure of the material. The production technology is detailed in projects [7-11].

The subject of research was a composition of alternating layers of the U8 carbon steel and austenitic 08H18n10 grade steel, whose samples dimensions are 10 mm x 10 mm. This project was focusing on assessing the influence of the structuration on the progress of chemical and thermal processing, so a composition was selected allowing for a decrease and preservation of the laminar layer from 100 μm to 2 μm without observing the recrystallization process as a result of technological cycles occurring.

Nitrogen was chosen as the saturating element. Chemical and thermal processing, nitriding was conducted in an ammonia gaseous environment NH_4 under the temperature of 540 $^\circ\text{C}$ for 45 hours.

3 Research results

The first technological cycle of producing multilayer metal materials allows for sample thickness of 100 μm and the number of layers equal to 100. Figure 1 shows the microstructure of the researched composition after nitriding [12-13].

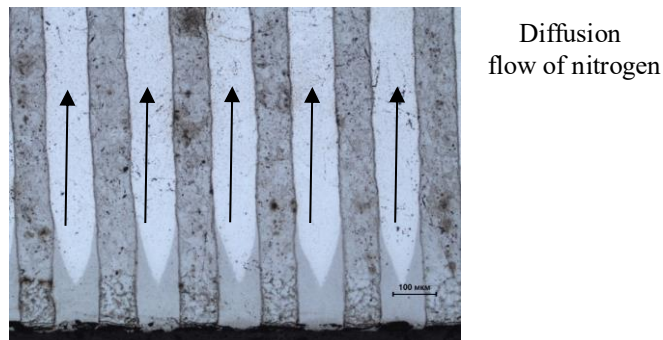


Fig. 1. Multilayer material microstructure U8+08H18n10 after nitriding, laminar layer thickness 100 μm .

The resulting microstructure shows that saturation leads to volumetric diffusion, as indicated by the nitrogen crust, after which grain boundary diffusion develops. While the grain boundary diffusion is occurring, the dopant only outflows into the austenitic 08H18n10 grade steel, due to the fact that carbon lowers the solubility of nitrogen, whereas chromium increases it (fig.2) [15], hence the nitrogen outflows from the boundaries specifically into the 08H18n10 steel layers.

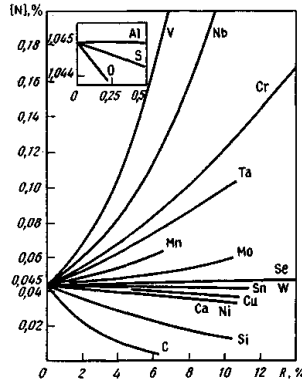


Fig. 2. The influence of alloying elements on the solubility of nitrogen in the steel [15].

The second technological cycle is completed by producing a 5 μm laminar layer, whose number of layers is equal to 2000. The sample has also undergone nitrogen saturation following the specified procedure.

The analysis of microstructure after the second technological cycle shows the same picture as after the first technological cycle. The volumetric diffusion occurs first and is followed by the outflow of the dopant into the 08H18n10 steel from the boundaries, which is also explained by increased solubility of nitrogen when chromium is present.

For a more detailed analysis, a microdiffraction analysis (EBSD) was performed on the selected composition after the second technological cycle with the laminar layer width of 5 μm with the number of layers equal to 2000. The analysis of the transition area (fig. 3), and the influence of nitrogen on the modification of the microdiffraction picture were especially interesting.

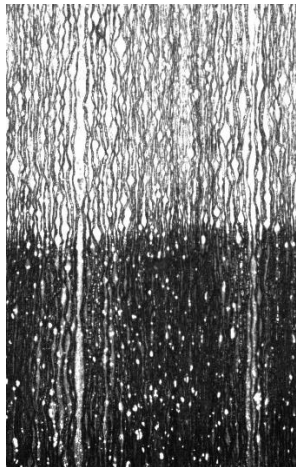


Fig. 3. U8+08H10n10 composition microstructure after the second technological cycle. Layer thickness – 5 μm , number of layers – 2000.

The microdiffraction analysis showed that the nitriding progress primarily spares the α -Fe phase (62%), which corresponds to the U8 carbon steel, whose layers are not penetrated by nitrogen on account of its low solubility, while the γ -phase is preserved at 38 %. This is explained by that fact that nitrogen diffusion happens primarily at the interlayer boundaries and is followed by the outflow from the 08H18n10 steel layer, which results into enrichment of those layers with nitrogen and recrystallization.

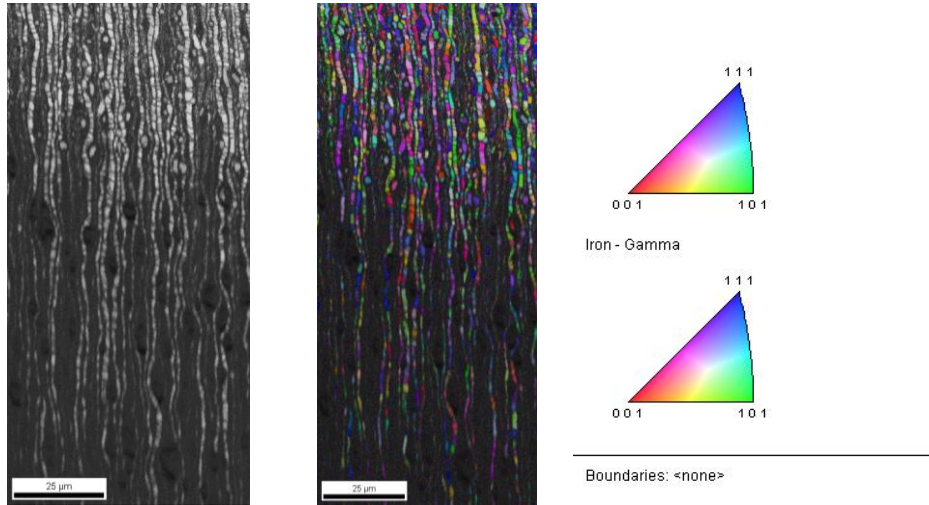


Fig. 4. U8+08H18n10 composition microdiffraction picture after nitriding.

4 Conclusion

Thus, we have reviewed the composition of multilayer materials where the laminar structure is preserved after the second technological cycle. Nitrogen alloying definitely changes the nature of the microdiffraction picture, resulting into the preservation of α -phase on account of the low solubility of nitrogen in it and recrystallization of the γ -phase during the active diffusion occurring in those specific layers of the composition.

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