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Analysis of Phase Transformations in Inconel 625 Alloy during Annealing

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The investigation focused on the characterization of the microstructure and chemical composition changes after annealing. The research was conducted on the Inconel 625 weld overlay deposited on a boiler steel. The annealing was performed for 10 h at temperatures from 600 to 1000 °C. The microstructure and chemical composition were examined by scanning electron microscope and transmission electron microscope equipped with an energy dispersive X-ray spectrometer. Weld overlays were produced by an innovative method of cold metal transfer.

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1. Introduction

Nickel-based alloys possess numerous specific properties, such as low thermal expansion, high electrical resistivity, and unique magnetic properties. However, the wide range and high concentration of alloying elements used in Ni-based alloys make the prediction of microstructure and properties of these alloys very difficult, especially when the alloys are subjected to high temperatures. Many alloys designed to be single phase actually exhibit compositions that are beyond the solubility limit and at elevated temperatures can form a wide range of secondary phases. This phenomenon occurs in two-phase $(\gamma + \gamma')$ superalloys, where brittle phases (such as σ, P, μ, Laves) with complex structures can form during long term, elevated temperature exposure [1]. These phases may form already during the solidification, especially at the final stage of the process due to the effect of microsegregation [1, 2].

Significant diversity in the chemical composition of nickel alloys bring on large differences in morphology and chemical composition of particular phases. The high content of alloying additions constitute a great challenge with regard to microstructure. There are difficulties in estimating the exact composition of a particular phase for specific contents of elements and at a certain temperature due to the lack of suitable phase equilibrium diagrams for complex systems. Multi-component alloys designed as a single-phase can contain more phases, since the solubility of the individual elements in the nickel alters in the presence of other elements. The precipitation of new phases may occur in alloys with molybdenum and niobium.

Intermetallic phases that may appear in nickel alloys:

— γ' Ni_3(Al, Ti) with a cubic crystal structure that is an ordered variation of face centered cubic structure [3],

— γ'' Ni_3(Nb, Ti) with a cubic crystal structure that is a variation of body-centered tetragonal structure [4].

Intermetallic topologically closed packed (TCP) phases: η Ni_3(Nb,Ti), δ Ni_3(Nb,Ti), σ A_xB_y-type, Laves A_2B-type, μ A_6B_7-type, G [3, 4].

Apart from the intermetallic phases carbides MC, M_6C, M_7C_3, $M_{23}C_6$, borides M_3B_2, chromium borides $M_{23}(C,B)_6$, nitrides and carbonitrides may be present in multicomponent nickel alloys [5, 6]. Carbides can be formed inside the grains or on grain boundaries in the form of large particles uniformly distributed throughout the alloy volume.

The solidification of nickel alloys results in formation of microstructure that is composed of directionally crystallizing dendrites. During solidification the natural chemical segregation occurs and the chemical composition of dendrites (cells) is different from the interdendritic spaces. Thus, within the interdendritic spaces another phase may form [7, 8].

Such elements like Nb, Mo, Si, and C have a high tendency to segregation [1, 9, 10]. They are pushed into the liquid during solidification, more and more enriching it. After completion of the solidification, these elements remain in the interdendritic spaces, where their concentration exceeds the average content in the alloy. Thus, in the interdendritic spaces oversaturated with these elements some intermetallic phases like σ phase can form. Such a situation occurs in alloys containing niobium and carbon. At the end of solidification NbC or other carbides as well as the Ni_3Nb phase can be formed in interdendritic spaces [11–13]. The tendency to segregation increases for silicon, the content of which should be limited in the alloy. Such elements like Fe, Cr, Co, and W slightly segregate to the liquid, and therefore the distribution is uniform.

The main objective of this research is to study precipitation phenomena occurring in a solid-solution strengthened Inconel 625 alloy subjected to annealing at elevated temperature.

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2. Methodology

The research was conducted on the Inconel 625 weld overlay deposited on a boiler steel. The choice of weld overlays as a research material allowed for an analysis of phase transformation in the areas of contact between two different materials (nickel alloy and steel), where the mutual diffusion of elements changes the chemical composition and enhances phase transformations. The deposited weld overlay solidifies rapidly, so its chemical composition deviates significantly from the equilibrium state.

The investigation focused on the characterization of the microstructure and chemical composition changes after annealing. The annealing was performed for 10 h at temperatures from 600 to 1000 °C. The microstructure and chemical composition were examined by scanning electron microscope (SEM) (FEI Inspect S50) and transmission electron microscope (TEM) (JEOL JEM-2010) equipped with an energy dispersive X-ray spectrometer.

Samples for research were delivered by SEFAKO — company specializing in the production of boilers. Weld overlays were produced by an innovative method of cold metal transfer (CMT). The CMT is a form of gas metal arc welding (GMAW) process, however, unlike the conventional GMAW, it is a fully controlled inverter welding process. To the key benefits of the CMT method belong very low heat transmission to the base metal, high process speed, very low spatter, as well as precision control of the welding arc and its stability. These CMT features allow for producing high quality weld overlays on steels [14, 15].

3. Results and discussion

The microstructure of the produced overlay was typical for conventionally cast metals. The areas adjoining to the fusion zone exhibited columnar dendrites and cell structure that emanates in the direction parallel to the heat extraction (Fig. 1). The solidification process resulted in a pronounced differences in chemical composition between dendrite cores and interdendritic spaces. The interdendritic spaces were enriched with Nb and Mo while the content of Ni and Cr in dendrites was higher than average. This is best illustrated on EDS maps (Fig. 2) that show segregation of the particular constituents.

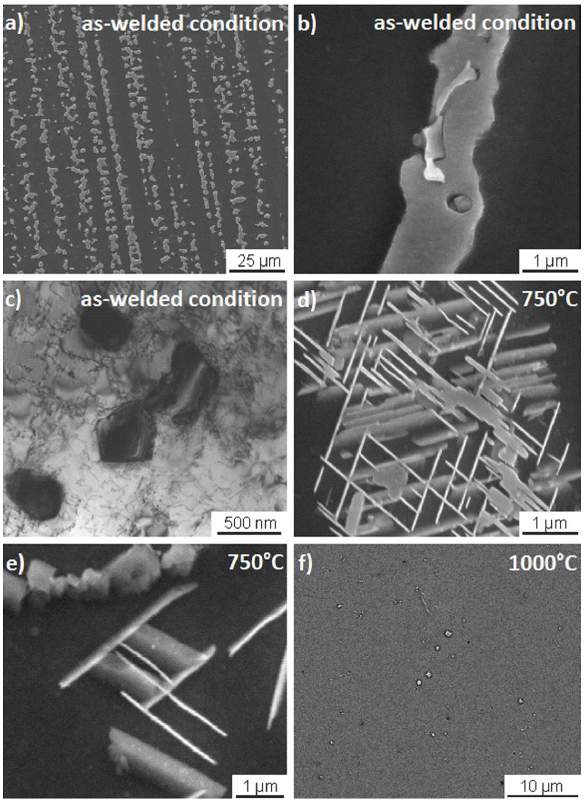

Fig. 1. The microstructure of weld layer: (a) dendritic structure, (b) Laves phase in the γ matrix, (c) carbide precipitates (TEM), (d,e) needle-like δ phase, (f) microstructure after annealing at 1000 °C.

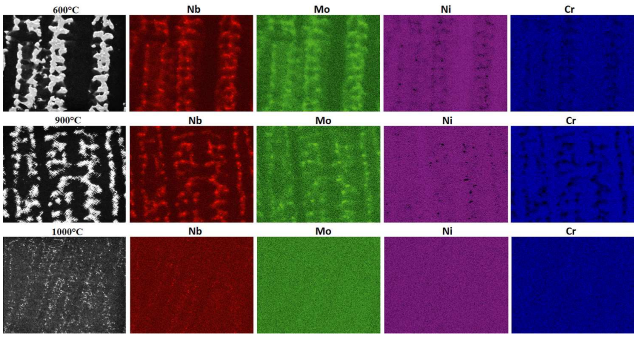

Fig. 2. Elemental distribution maps Nb, Mo, and Ni in the area presented in SEM image.

Figure 1a–f shows the changes in microstructure produced by the annealing at different temperatures. Between 600 °C and 700 °C no changes were observed in microstructure. However, it was found that above 750 °C, the secondary phase dissolves and the orthorhombic intermetallic δ phase forms. This phase also exhibits the Ni_3Nb stoichiometry but its morphology changes to the needle-type form, as it is shown in Fig. 1d–e. After annealing at 1000 °C (Fig. 1f) no differences in chemical composition were found between the dendrite cores and the interdendritic spaces. The needles observed previously in the interdendritic spaces disappeared. The EDS line distribution of main elements in the weld overlay for temperatures 600 °C and 1000 °C is shown in Figs. 3 and 4. It is apparent that no difference in chemical composition between the dendrite cores and the interdendritic spaces after annealing at 1000 °C.

The results of chemical composition analysis for the precipitates of other phases shown in Fig. 1 are presented in Table I. In the as-welded condition, two types of precipitates were observed: MC-type carbides (Fig. 1c) enriched with Nb and Ti, and likely a Laves phase enriched with Nb and Mo. Above 750 °C, the δ phase was observed (Fig. 1d–e) and probably M_6C-type carbide. Both phases were enriched with Si occurring along the grain boundaries.

TABLE I

The chemical composition of γ phase, carbides, δ phase and Laves phase in the weld overlay.

Phase	Elements composition [wt.%].						
	Nb	Mo	Cr	Fe	Ni	Ti	Si
γ	3	9	22	1	65	–	–
MC	60	11	6	1	13	9	–
Laves	23	16	13	1	47	–	–
δ	25	7	3	1	63	1	–
M_6C	13	9	13	2	35	1	27

TABLE II

Calculated values of k for Nb, Mo, Cr, Fe, and Ni in the weld overlay.

Parameter	Elements composition [wt.%].				
	Nb	Mo	Cr	Fe	Ni
C_{core}	2.5	9.0	22.5	0.5	65.5
C_0	4.3	9.9	22.1	0.5	65.2
k	0.58	0.91	1.02	1.00	1.04

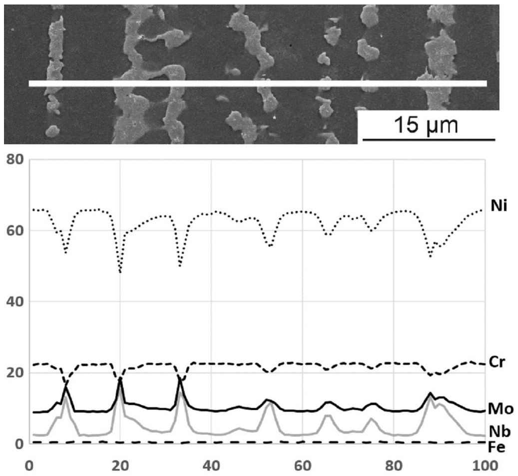

Fig. 3. Line distribution of chemical elements in weld overlay (600 °C).

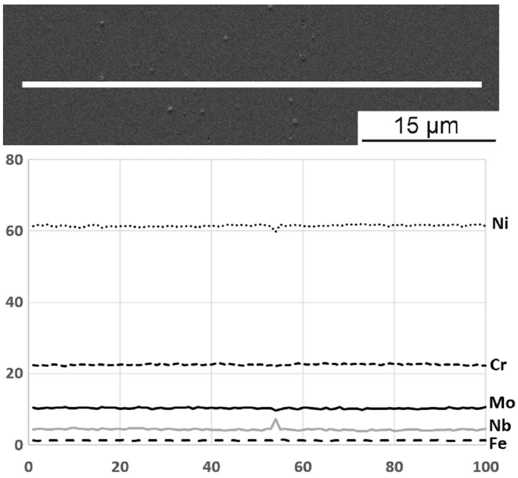

Fig. 4. Line distribution of chemical elements in weld overlay (1000 °C).

Based on the results of chemical analysis of the dendrite cores and the average composition of the weld overlay, value of the parameter k was determined for the major elements. Table II shows the average concentrations of Nb, Mo, Cr, Fe, Ni and the calculated k coefficient. The results confirm that Nb and Mo ($k < 1$) segregate to the interdendritic spaces. By contrast, Ni and Cr ($k > 1$) segregate to the dendrite cores [1, 6, 9].

4. Conclusions

- Microstructural examination of weld overlay revealed characteristic dendritic structure in as-overlaid samples.
- The solidification process resulted in a pronounced differences in chemical composition between dendrite cores and interdendritic spaces. The interdendritic spaces were enriched with Nb and Mo.
- The microstructure depended on the annealing temperature. Between 600 °C and 700 °C no changes were observed in microstructure. Above 750 °C, the secondary phase dissolves and the orthorhombic intermetallic δ phase forms. After annealing at 1000 °C, the needles observed in the interdendritic spaces have disappeared.
- The presence of MC-type and M_6C-type carbides in the weld overlays were also observed.

Acknowledgments

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