

### M. ROZMUS-GÓRNIKOWSKA\*, Ł. CIENIEK\*, M. BLICHARSKI\*, J. KUSIŃSKI\*

## MICROSTRUCTURE AND MICROSEGREGATION OF AN INCONEL 625 WELD OVERLAY PRODUCED ON STEEL PIPES BY THE COLD METAL TRANSFER TECHNIQUE

# MIKROSTRUKTURA I MIKROSEGREGACJA SKŁADU CHEMICZNEGO NAPOIN ZE STOPU INCONEL 625 NAPAWANYCH TECHNIKĄ CMT NA RURY KOTŁOWE

The aim of this work was to investigate the development of microstructure and variations in chemical composition in commercial Inconel 625 coatings on a ferritic-pearlitic steel overlaid by the CMT method.

The investigation showed that microsegregation occurring during the weld overlay solidification makes the dendrite cores to be richer in Ni, Fe and Cr and the interdendritic regions in Mo and Nb. Niobium shows the strongest tendency to segregation during solidification; molybdenum tends to segregate less and chromium has the lowest tendency to segregation. Although Inconel 625 is a solid solution strengthened alloy, Nb and Mo-rich phases are formed in the interdendritic regions of weld overlays.

Keywords: microstructure, microsegregation, Cold Metal Transfer (CMT), boiler pipes Inconel 625

Celem pracy była ocena mikrostruktury i składu chemicznego powłok napawanych metodą CMT na podłoże ze stali ferrytyczno-perlitycznej, jak również ocena mikrosegregacji pierwiastków stopowych następująca podczas krystalizacji napoiny. Badania wykazały, że w wyniku mikrosegregacji zachodzącej w trakcie krystalizacji napoin rdzenie dendrytów bogatsze są w Ni, Fe i Cr, natomiast obszary międzydendrytyczne w Mo i Nb. Podczas krystalizacji najsilniej segreguje niob, w mniejszym stopniu molibden, natomiast najmniej segreguje chrom. Pomimo, że Inconel 625 jest stopem umacnianym roztworowo, to w przestrzeniach międzydendrytycznych napoin tworzą się fazy bogate w Nb i Mo.

### 1. Introduction

Waste burning furnaces produce gases containing very aggressive chlorides and fluorides that exert a negative impact on furnace elements like heat exchanger pipes and combustion chambers. Thus, these components require effective protection against corrosion and erosion. At the present, the operational durability of the furnace elements that are most exposed to corrosion is increased by overlaying the working surfaces with nickel-based alloy coatings. Most often, Inconel 625 alloy is used in the overlaying process. This alloy is characterized by very good creep resistance at high temperatures, as well as corrosion resistance in aggressive environments [1,2].

The principal requirement for nickel-based overlays is low iron content (below 5 wt %) in the external overlay zone. Simultaneously, the coating thickness should not exceed 2.5 mm. In order to reduce the Fe content in a weld overlay, a grade of Inconel alloy containing less than 0.5 wt %. of Fe is used in the overlaying process. However, the Fe content in the overlaid increases due to melting of the substrate material (steel) and its dissolution in the overlaid coating. It was found that if the Fe content and, in general, chemical heterogeneity in the weld overlay increases, the corrosion resistance of the overlay decreases [3,4]. The chemical heterogeneity results from the solidification process and is manifested by dendritic segregation (microsegregation). The intense microsegregation – primarily of such elements as Nb and Mo – leads to the formation of secondary phases and, as a consequence, reduces the corrosion resistance of the overlaid alloy [5÷7].

Commonly, the following welding methods are used to deposit overlay coatings: gas welding (flame welding), arc welding, laser beam or electron beam welding. The process of overlaying with pulsed wire feeding, referred to as Cold Metal Transfer (CMT) process, is a novel overlaying method that introduces a considerably smaller amount of heat into the material, compared to traditional arc welding methods [8].

The aim of this work was to investigate the development of microstructure and variations in chemical composition in commercial Inconel 625 coatings on a ferritic-pearlitic steel overlaid by the CMT method.

#### 2. Material and experimental procedure

Boiler pipes, made of 16Mo3 Grade steel and weld overlaid with Inconel 625 by the CMT method, were investigated in this study. The Fe content in the Inconel used in the over-

<sup>\*</sup> AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY, FACULTY OF METALS ENGINEERING AND INDUSTRIAL COMPUTER SCIENCE, AL. A. MICKIEWICZA 30, 30-059 KRAKOW, POLAND

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laying process was about 0.3 wt %. The overlaid coating was 2.3 mm thick, on average.

Metallographic cross-microsections of the overlaid pipe were selected for the examination of microstructure, chemical composition and dendritic microsegregation. The microsections were parallel to pipe axis and perpendicular to the pipe surface. The etching process was performed in two stages. First, the substrate was chemically etched in a 2% solution of nitric acid in C<sub>2</sub>H<sub>5</sub>OH. Then, the coatings were electrolytically etched at room temperature in a 10% solution of CrO<sub>3</sub>. In the etching process, the voltage of 2 V was applied for 15 s.

The microstructure examinations were carried out with an Axio Imager MAT. M1m light microscope (LM) manufactured by the Carl Zeiss company and with a Hitachi S-3500N scanning electron microscope (SEM). Analyses of the chemical composition, qualitative and quantitative, aimed at determining variations in the content of Ni, Fe, Cr, Mo and Nb along the direction perpendicular to the surface of the examined coatings and at revealing of dendritic microsegregation, were carried out by an energy X-ray dispersive spectroscopy (EDS) on the scanning electron microscopes: a FEI NanoSEM 450 equipped with an EDAX EDS detector and a Hitachi S-3500N microscope equipped with a Noran EDS detector. In order to determine variations in the content of Ni, Fe, Cr, Mo and Nb in the direction perpendicular to the surface of overlaid coatings spot analyses were performed in successive measurements. Point to point analyses were performed every few  $\mu m$ across the entire partially mixed zone. The acquired results provided the basis for the preparation of diagrams that show the dependence of the content of the elements as a function of the distance from the fusion boundary.

Analyses along straight lines perpendicular to the direction of dendrite growth were also performed. These measurements allowed for revealing the segregation of alloy elements occurring during overlay solidification. The analysis was performed over several neighboring dendrites. The acquired results provided the basis for the construction of diagrams showing the distribution of major elements, i.e. Ni, Fe, Cr, Mo and Nb (wt %) in micro areas and thus illustrating their segregation during solidification.

### 3. Results and discussion

The microstructure of the weld overlaid pipe used in this investigation is presented in Fig. 1. The following zones were clearly distinguished in the overlaid pipes: a fusion zone, an un-etched partially mixed zone, a partially melted zone, a heat-affected zone and the base material. A detailed discussion of the zones found in overlaid pipes is presented elsewhere [9]. The overlays exhibited a uniform fusion boundary. According to literature, the microstructure near the fusion boundary in dissimilar welds depends on crystal structure of the heat affected zone in the base metal and crystal structure of the overlay. In the case when the base metal is ferritic and the overlay is austenitic, the normal epitaxial growth may be suppressed. This can result in the formation specific boundaries, referred to as the Type II boundaries, that run roughly parallel to the fusion boundary. At distances farther from the heat affected zone, the cellular-dendritic microstructure was observed. The direction of dendrites coincides with the direction of heat transfer during solidification (Fig. 2). The pronounced changes of the weld overlay microstructure were found in the zones where successive welding beads overlapped. It is believed that the overlay solidification in these zones started on the partly-fused former bead. Moreover, the preferred crystal orientations "inherited" from previous beads were observed (in this case, both beads had the same FCC crystal structure).



Fig. 1. Microstructure of the weld overlay with a marked microstructural zone (LM)



Fig. 2. Microstructure of the weld overlay near the fusion boundary (SEM)

The distribution of Ni, Fe, Cr, Mo and Nb (wt %) as a function of the distance from the fusion boundary is shown in Fig. 3. The thickness of zone, where the deposited material is not fully mixed with the substrate, i.e. the partially mixed zone, was about tens  $\mu$ m. The chemical composition of this zone clearly differs from the chemical composition of the substrate material and the chemical composition of the deposited material. The Fe content in the partially mixed zone decreased while the Ni, Cr, Mo and Nb contents increased with the distance from the fusion boundary. Near the overlay surface, at the distance of about 2.3 mm from the fusion boundary, the Fe content was about 3% wt. The increased Fe content in the partially mixed zone and in the coating resulted from the melting and dissolution of the substrate material (steel).

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Fig. 3. Distribution of Fe, Ni, Cr, Mo, Nb (wt %) as a function of the distance from the fusion boundary



Fig. 4. Microstructure of the weld overlay (a) and the line profile of the Ni, Cr (b) and Mo, Nb, Fe (c)(wt %) along the dashed line

Figure 4 shows typical overlay microstructure together with the reference line along which the chemical analysis was

performed. This analysis allowed for revealing and assessing the degree of segregation of elements resulted from the solidification process. These figure also shows diagrams of the content (wt %) of Ni, Cr, Fe, Mo and Nb as a function of length. The chemical analysis showed that the cores of dendrites are richer in Ni, Fe and Cr, while the areas between dendrite arms, i.e. interdendritic regions, are richer in Mo and Nb. Dashed lines are drawn on the diagrams in the areas corresponding to the interdendritic regions, where the contents of Nb and Mo reached the highest values.

It has already been established that the distribution of a particular element during solidification of nickel alloys depends primarily on the value of quantity, referred to as the distribution coefficient k [5, 6]. If the value of k for an element is lower than 1, the content of this element in the solid phase is lower than in the liquid one. It means that the concentration of this element within a dendrite is lower than in the areas that solidify as the latest. If the value of k is higher than 1, the concentration of the element in the solid phase is higher than in the liquid phase and the segregation process proceeds in the opposite direction, i.e. the concentration of this element within a dendrite is higher than in the areas that complete solidification. Fe, Cr and Ni belong to elements that tend only slightly to segregate in Inconel 625 alloy, because - according to the relevant literature [5,6] – the values of the distribution coefficient k for Fe, Cr and Ni in Inconel 625 alloy are only slightly higher than 1 ( $k_{Fe} = 1.02$ ;  $k_{Cr} = 1.05$ ;  $k_{Ni} = 1.04$ ). The value of k for Mo is lower than 1 ( $k_{Mo} = 0.86$ ). Therefore, Mo tends segregate to the liquid and, once alloy solidification is finished, the interdendritic regions are considerably enriched with Mo. The value of the distribution coefficient for Nb is lower than for Mo ( $k_{Nb}$  =0,50). Therefore, among the analyzed elements, Nb has the strongest tendency for segregation and the interdendritic regions are considerably richer in this element, once solidification is completed. The results of the linear analysis of chemical composition confirmed that Nb has the strongest tendency to segregate. The presented results confirm also earlier results obtained in similar investigations [5÷7].

Although Inconel 625 is a solid solution strengthened alloy and, as such, it should be homogenous with respect to chemical composition and microstructure, some precipitates were found in the interdendritic spaces. Two types of precipitates were identified: elongated one and minute characterized by sharp-edged, angular shapes. Both phases were formed within the interdendritic areas of weld overlays (Fig. 5).

### 4. Conclusions

- The CMT method can be used to produce a defect-free weld overlay characterized by low content of iron in the surface zones of the overlay.

 Microsegregation occurring during the weld overlay solidification makes the dendrite cores to be richer in Ni, Fe and Cr and in the between dendrite arms in Mo and Nb.

– Niobium shows the strongest tendency to segregation during solidification; molybdenum tends to segregate less and chromium has the lowest tendency to segregation.







– Although Inconel 625 is a solid solution strengthened alloy, Nb and Mo-rich phases are formed in the interdendritic areas of weld overlays.

### Acknowledgements

The project was financed by National Science Center; grant number DEC-2011/01/D/ST8/04948.

Received: 20 March 2014.

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