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MICROSTRUCTURE, CHEMICAL AND PHASE COMPOSITION OF CLAD LAYERS OF INCONEL 625 AND INCONEL 686

The article presents the investigation results of microstructural, chemical composition changes after clad layers of Inconel 625 and Inconel 686 alloys weld overlaid on boiler tubes. The boiler tubes made of 16Mo3 steel were clad in the Boiler Factory SEFAKO S.A. using CMT technique. The microstructure and chemical composition of coatings were analysed using light microscopy, scanning and transmission electron microscopy. The value of the partition coefficient k was determined for Inconel 625 and Inconel 686 clad layers. The value was calculated by dividing the average content (% wt.) of Ni, Cr, Mo, Fe, Nb and W.

Keywords: Inconel 625, Inconel 686, SEM, EDS, TEM

1. Introduction

Protective coatings are widely used in all modern power generation systems, including utility turbines for electric power production and gas turbines, since their improved efficiencies, lower emissions and reduced downtime of plant and can be achieved with the higher temperature and corrosive resistances.

Nowadays, Ni-base alloys are typically used to manufacture engineering components, or coatings for protection of cheaper metallic substrates, which should work in extreme conditions including mechanical loads and an aggressive environment at high temperature. Due to the excellent high temperature corrosion resistance and good strength at high temperatures Ni-base alloys can work in aggressive environments [1,2]. This Ni-base superalloys are widely used in several industrial sectors, like chemical and petrochemical plants, power generation sector [3,4], aircraft engines components. Inconel 625 as well as Inconel 686 are used in different applications because of its strength, excellent fabricability and outstanding corrosion resistance. Inconel 625 and Inconel 686 can be applied also in power boilers in the waste incineration plants. Because of the high cost of Ni-base superalloys compared to stainless steels, they are reserved for these particular cases where the stainless steels are not suitable or when the purity or safety are critically important [5,6]. This is the reasons, to reduce components' costs by using Ni-base superalloys as coatings to protect cheaper materials.

Among a variety of hardfacing techniques, cladding is another form of surface treatment, where the bulk material surface is given as a protective layer of another material [7]. The clad layers have more superior properties than those of the

bulk material. Chemical composition of the clad layer should be homogenous and the concentration of Fe entering from the base material (steel) into the coating should be as low as possible [8].

To perform the Ni-base clad layers, without introducing too much Fe, a new weld technique called Cold Metal Transfer (CMT) was used. The cold metal transfer process which was invented by Fronius company as a modified Metal Inert Gas (MIG) welding process [9]. CMT is the method of submerged arc welding with a completely new way of droplet avulsion from the wire detachment by means of the wire-motions incorporated in the digital process-control. In comparison with conventional Gas Metal Arc (GMA) process, CMT reduces the thermal input by achieving almost current-free metal transfer from the wire to the coating. Indeed, this reduces the Fe content (coming from the remelted substrate) in a clad overlay. Characteristic and very important for a CMT method is that the coating is received at lower temperature [10]. Even that, using this technique, good metallurgical bonding can be obtained between coating and substrate.

The clad layers, like dissimilar welds, consist of the four distinct microstructural zones: fusion zone (*FZ*), partially mixed zone, partially melted zone and heat affected zone (*HAZ*). Between partially mixed zone and partially melted zone is located fusion boundary, which separates base material from clad layers (Fig. 1) [11].

Ni-base superalloys, like Inconel 625 and Inconel 686, were clad on 16Mo3 boiler tube steel by using CMT technique. For a detailed analysis of the microstructure clad layers advanced techniques like: light microscopy (LM), scanning electron mi-

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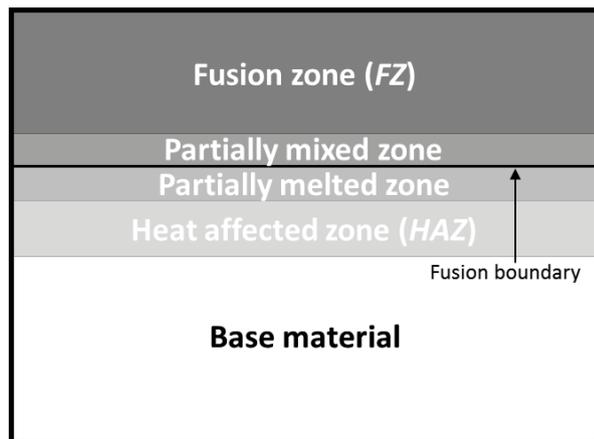


Fig. 1. Characteristic microstructural zones in dissimilar welds

croscopy (SEM) transmission electron microscopy (TEM) were applied. Chemical composition of the clad layers were analysed by energy dispersive X-ray spectroscopy (EDS).

2. Materials and method

For investigations the samples were cut from original boiler tubes. The boiler tubes were made of the 16Mo3 steel and clad with nickel-based alloys like Inconel 625 and Inconel 686 using Cold Metal Transfer (CMT) technique. The cladding process was carried out in Boiler Factory SEFAKO S.A. In the CMT process the wire is moved towards the work piece (boiler tube) until a short circuit occurs. When the short circuit occurs the wire speed is reversed and the wire is pulled back. After then short circuit opens another time, the wire speed is contrary, the wire moves towards the work piece once again and the process reiterates. The cladding process was conducted at a cladding power voltage 20 V, and cladding current 200 A. Gas flow rate was 17 l per minute, wire feed speed was 0.8 meter per minute, the peripheral speed of the tube was 4.5 rotation per minute and diameter of the stitch was ratio 2.8 mm. The chemical compositions of the investigated Inconel 625, Inconel 686 alloys and 16Mo3 boiler steel tubes are shown in Table 1 [12,13]. On the boiler tubes (\varnothing 80 mm) was applied only one layer of the clad layer (Inconel 625 and 686). The thickness of the clad layers was approximately 2.5-3.0 mm.

TABLE 1

Chemical composition of Inconel 625, Inconel 686 wires used for cladding, and boiler tubes 16Mo3 substrate (% wt.)

Alloy	Ni	Cr	Mo	Nb	W	Fe	C	Mn	Si	Ti
Inconel 625	Bal.	23.2	9.4	3.4	—	0.3	0.1	0.4	0.4	0.4
Inconel 686	Bal.	20.2	15.0	—	4.3	0.1	0.05	0.35	0.03	0.2
16Mo3	—	0.3	0.3	—	—	Bal.	0.16	0.65	0.35	—

The light microscope examinations on longitudinal cross-section of clad layers were performed by Axio Imager Mat. M1m Carl Zeiss microscope. The study of clad layers

microstructure and chemical composition were performed by FEI Nova NanoSEM 450 equipped with an energy dispersive X-ray spectrometer (EDS) (EDAX Company). The specimens for microstructure and chemical composition investigations were out perpendicular to the clad layers surface were subjected to two stage etching. The base metal (boiler tube steel 16Mo3) was chemically etched in 2% solution of nitric acid in C_2H_5OH , after that the Inconel 625 coatings were electrolytically etched in 10% water solution of CrO_3 at a preset voltage of 1.8-2.0 V. Inconel 686 coatings was electrolytically etched in cold ($10^\circ C$) methanol 25% solution of HNO_3 at a preset voltage of 1.5 V. The changes of the elements distribution were performed using the point EDS analysis perpendicular the surface of the clad layers.

Specimens for TEM investigations were prepared from 1 mm slices taken from the clad layers of Inconel 625 and Inconel 686. The slices were first ground mechanically to a thickness of about 0.7-0.10 mm. 3 mm disc were subsequently punched from these grounded samples and dimpled on each side and thinned by the dual jet electrolytic polishing technique. Electrolytic polishing was carried out at 30 V in a medium containing one part of perchloric acid and four parts of ethanol. During electrolytic polishing temperature of the bath was maintained at $-30^\circ C$.

The value of the partition coefficient k was determined for Inconel 625 and Inconel 686 clad layers. The value was calculated by dividing the average content (% wt.) of Ni, Cr, Mo, Fe, Nb and W (attempt of twenty measuring points) in dendrite cores (C_3) by average content in weld overlay (C_0), which were measured by energy dispersive X-ray spectroscopy (EDS). If the obtained partition coefficient k is lower than 1 ($k < 1$) major elements segregate during solidification to interdendritic spaces. By contrast, if this coefficient is greater than 1 ($k > 1$), elements segregate to dendrite core. When the coefficient k equals to 1 ($k = 1$) segregation phenomenon does not occur.

3. Results and discussions

The light microscopy investigations of boiler tubes with the clad layers are shown in Fig. 2. An Inconel 625 and Inconel 686 coatings clad on boiler tubes are shown in Fig. 2. The light microscopy investigations showed that the clad layers were free from spatter, porosity, cracking, incomplete fusion or other defects. The clad layers had good adhesion to the steel surface. The following zones were clearly distinguished in the overlaid tubes: a fusion zone (FZ), an un-etched partially mixed zone, a partially melted zone, a heat affected zone (HAZ) and the base materials. The clad layers exhibited a uniform fusion boundary. The thickness of the partially mixed zone of each welded clad layers was within the range is approximately 10 μm for Inconel 625 and 5 μm for Inconel 686 (Fig. 4).

The microstructure of the base steel in a close vicinity to the fusion line – in the heat affected zone, was in both steels the ferritic-bainitic. Both steels had the ferritic-pearlitic microstructure in areas lying far away from fusion line.

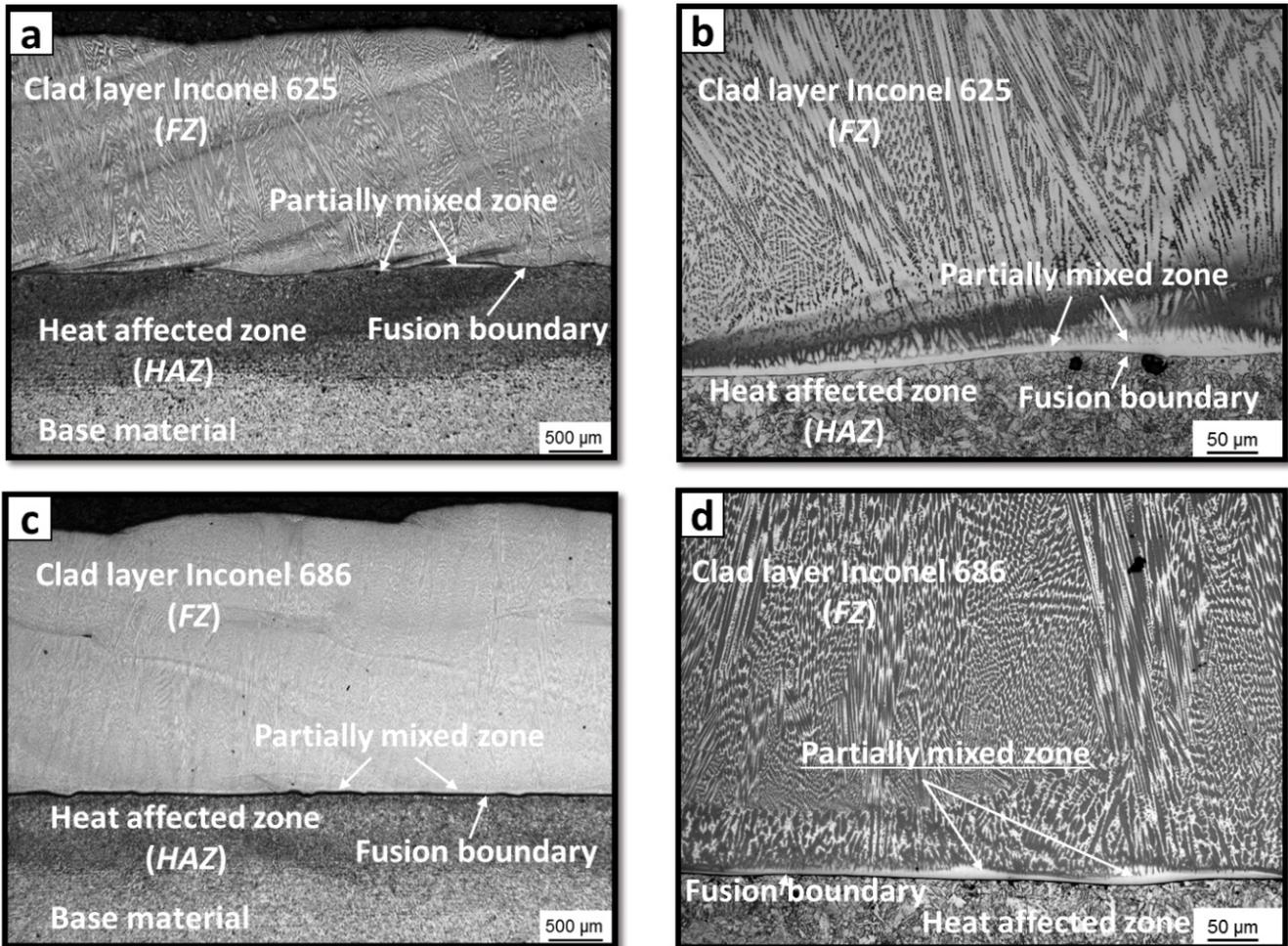


Fig. 2. Microstructure of: Inconel 625 (a,b); Inconel 686 (c,d); clad layers obtained on 16Mo3 boiler tube

Fig. 3 shows microstructure of the central areas of Inconel 625 (Fig. 3a) and Inconel 686 (Fig. 3b) the clad layers fusion zone. The cellular-dendritic microstructure was observed in the both clads layers. The dendrites arms were parallel to the direction of the heat flow.

Based on the results of chemical analysis, the partition coefficient k was calculated for the major elements of Inconel 625 and Inconel 686 clad layers. These analyses were performed in central areas of fusion zone. Table 2 shows contents of particular elements in dendrite cores (C_s), the average content of these

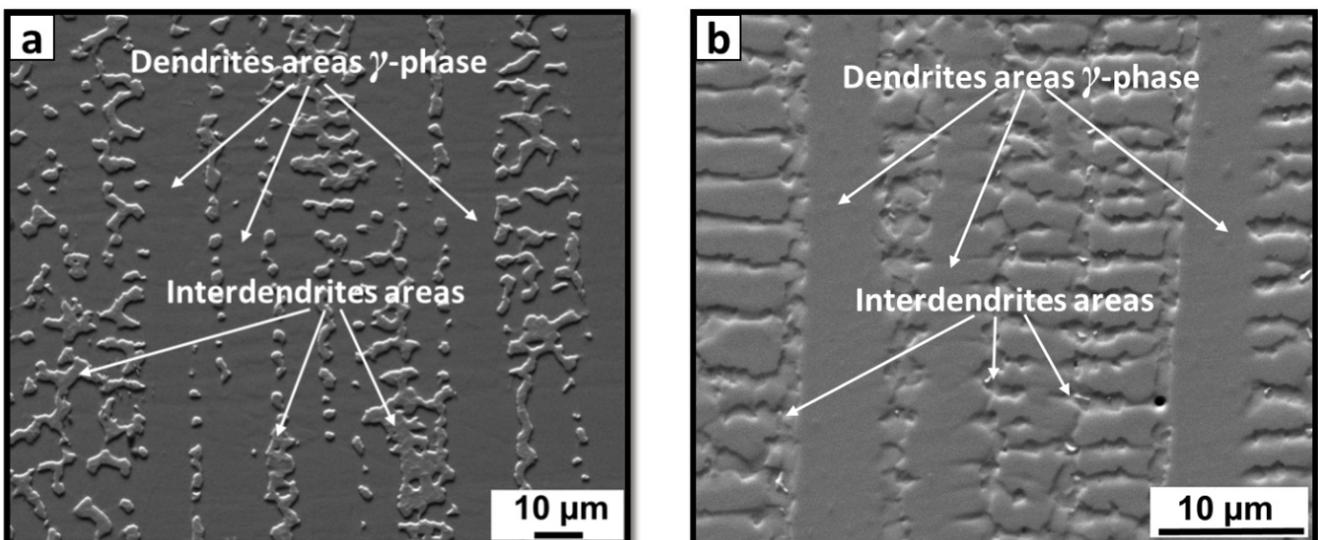


Fig. 3. Microstructure of fusion zone: a) Inconel 625, b) Inconel 686 the clad layers

elements in the clad overlay (C_0) and the values of the partition coefficient k .

For Inconel 625 clad layers, the values of the partition coefficient calculated for Nb, Mo, Cr, Fe and Ni. The results indicated a strong tendency to segregation of each elements during solidification. The values of the coefficient calculated for Nb and Mo were 0.49 and 0.86 respectively. While the values designated for Cr, Fe and Ni were higher than 1 1.03, 1.10 and 1.05 respectively. Since resulting values for Nb and Mo are lower than 1, these elements segregate during solidification to interdendritic spaces. By contrast Cr, Fe and Ni segregate to dendrite cores.

For Inconel 686 k coefficient was calculated for the same elements like for Inconel 625 but with the exception of Nb, which was replaced by W. Just as like in Inconel 625 alloy with Nb, in Inconel 686 clad layers the results indicate a strong tendency to segregation in particular Mo. The value of k coefficient was for Mo 0.78. The tendency to segregate also exhibited Cr and Fe, which coefficients k was 0.97 and 0.88 respectively. The partition coefficient of W ($k = 1.04$) indicates that this element does not segregate to the interdendritic spaces or occurs a small segregation to the dendrites cores. The value of the coefficient k calculated for Ni was 1.08. The obtained results are compatible with those found in the relevant literature [14,15].

Fig. 4 and Table 3 present the EDS point analyses for Inconel 625 and Inconel 686 fusion zone, non-etched partially mixed zone and heat affected zone. The Fe content is clearly higher in the partially mixed zone than in the both coatings, 56% Fe in the case of Inconel 625 and 51% Fe of Inconel 686 respectively. The Fe amount decreases with the distance from the (HAZ/FZ) interface towards the coatings surface (Table 3). Was observed that the amount of Fe is much decreases with the distance from the interface towards the coating surface. The EDS analysis showed that of Ni, Cr, Mo, Nb in Inconel 625 and W of Inconel 686 is much lower concentration than in the coatings and it increases gradually with distance from the interface.

TABLE 2

The partition coefficient (k) for nickel alloys: Inconel 625 and 686

Inconel 625					
Element	Nb	Mo	Cr	Fe	Ni
C_s	2.45	8.37	23.43	1.37	64.39
C_0	5.04	9.70	22.70	1.25	61.32
k	0.49	0.86	1.03	1.10	1.05
Inconel 686					
Element	W	Mo	Cr	Fe	Ni
C_s	4.97	13.93	19.49	0.35	61.26
C_0	4.78	17.93	20.08	0.40	56.83
k	1.04	0.78	0.97	0.88	1.08

TABLE 3

Chemical composition of the analysed zones (% wt.)

Zone	Alloy	Ni	Cr	Mo	Nb	W	Fe
Fusion zone (FZ)	Inconel 625	60.9	22	8	2.2	—	6.9
	Inconel 686	55.5	19	12	—	3.4	10.1
Mixed partially zone	Inconel 625	29.8	10.4	2.5	1.3	—	56
	Inconel 686	30.6	10.3	6.3	—	1.8	51

The TEM investigations of clad layers (Fig. 5) clearly show the presence of four secondary phases observed in the γ – fcc matrix.

Based on the studies of J.N. DuPont [16,17] the solidification process of Inconel 625 alloy begins with the formation of γ (gamma) phase. In the clad layers can also form MC carbides and the Laves phase [15-17].

The occurrence of γ phase was also confirmed by SEM observations (Fig. 3). The TEM bright-field image in Fig. 5 shows the interdendritic areas where the rod-shaped particle were recognized. This particle was identified as the Laves phase. In the clad layers of Inconel 625 the occurrence of MC carbides was also revealed (Fig. 5c). The phase identification was performed with the SAED analysis and revealed the occurrence of

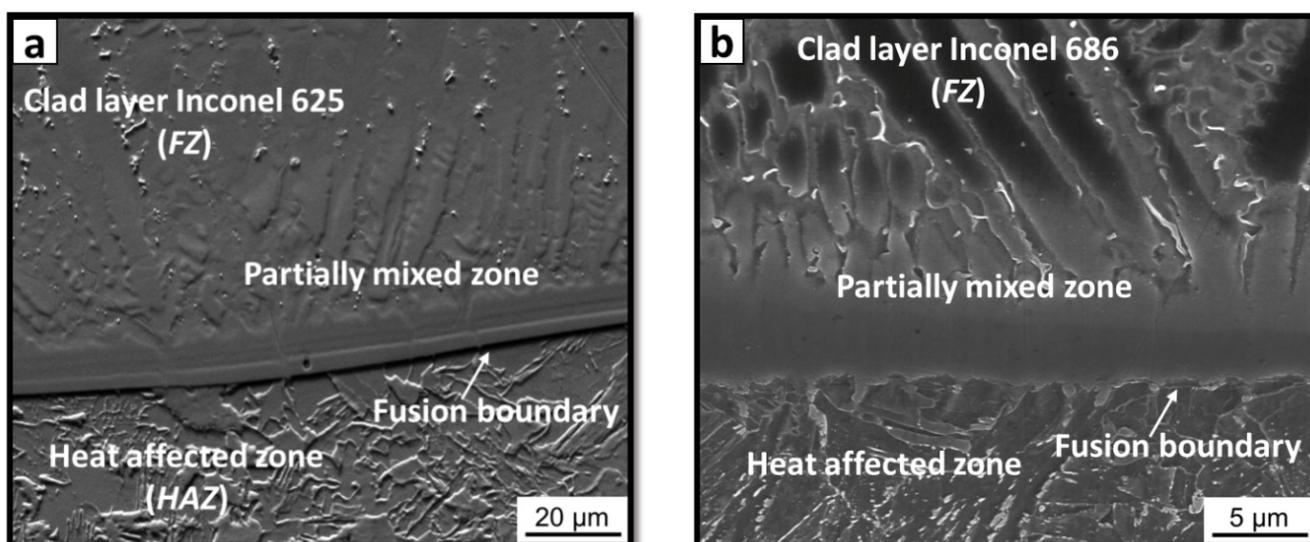


Fig. 4. Microstructure of the clad layers with a marked partially mixed zone in: a) Inconel 625, b) Inconel 686

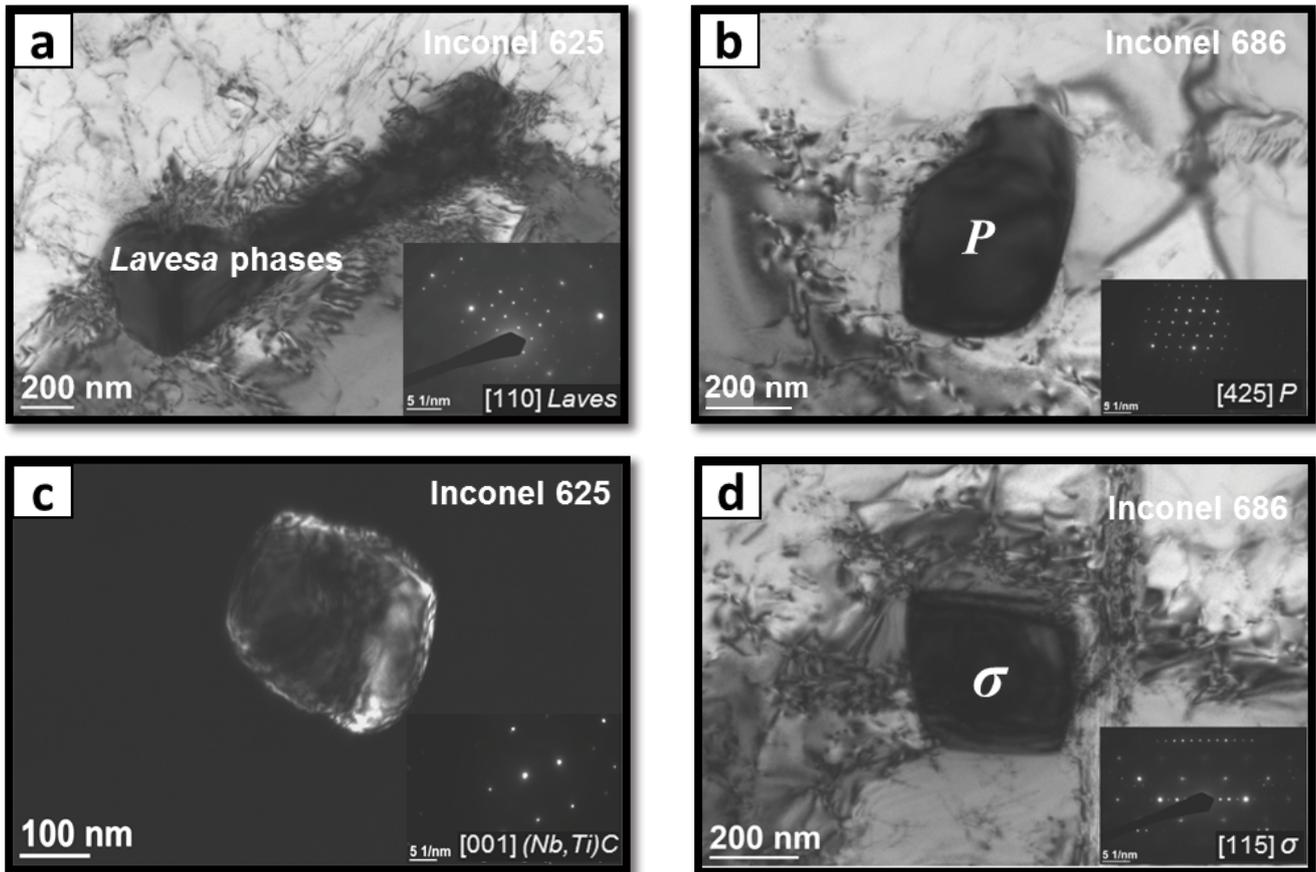


Fig. 5. TEM images showing the TCP phase precipitate observed in the interdendritic area of clad layers: a) Inconel 625 (BF), b) Inconel 686 (BF), c) Inconel 686 (DF), d) Inconel 686 (BF)

(Nb, Ti) C phase in [001] zone axis (Fig. 5). The microstructural observations showed that around MC carbide the dislocations were located (Fig. 5c).

During the solidifications process of clad layers, liquid enriched in Mo and W in Inconel 686, whereby in the final stage of the TCP phase (Topologically Close Packed) to exude like P or σ .

The TEM bright field images (Fig. 5b,d) show the presence of the precipitates with crystalline structure it was found diffraction pattern corresponds to the P -phase and a σ phases. In the clad layers of Inconel 686 many defects were observed. The J.N. DuPont et al [16-18], suggested that the secondary phase formed at the end of solidification is σ -phase, which subsequently becomes P phase as a results of solid state transformation.

4. Conclusion

LM and SEM studies showed that Inconel 625 and Inconel 686 clad layers have the characteristic cellular-dendritic microstructure. The cellular-dendrites solidified in a direction parallel to the direction of the heat transfer during solidification of clad layers. In the clad layers of Inconel 625 the γ phase, (Nb, Ti)C and the Laves phase were observed. The P -phase and σ -phase were observed in clad layers of Inconel 686. EDS analysis revealed the microsegregation of Nb and Mo into the cellular-dendritic

microstructure of Inconel 625 clad layers and microsegregation Mo, Cr and Fe in the clad layers of Inconel 686. The Fe content is clearly higher in the partially mixed zone than in the fusion zones and decreases with the distance from the interface HAZ/FZ towards the coating surface.

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