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MICROSTRUCTURAL EVOLUTION OF INCONEL 625 DURING THERMAL AGING

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Inconel 625 is due to alloying elements prone to precipitation of different intermetallic phases and secondary carbides during thermal aging. The base of investigation is nickel superalloy Inconel 625 in hot rolled state. Thermal aging was conducted at temperature 650 °C with different duration of treatment for each sample. Microstructural analysis was performed by light microscope and scanning electron microscope. The results of microstructure observation showed the precipitation of intermetallic γ››- Ni_3Nb phase in the γ matrix and δ-Ni_3Nb phase with $M_{23}C_6$ secondary carbides at the grain boundaries.

Key words: inconel 625, thermal aging, precipitation, intermetallic phases, secondary carbides

INTRODUCTION

Inconel 625 is a solid solution strengthened nickel superalloy, by addition of chromium, molybdenum, niobium and other minor elements [1,2]. Due to these elements and minor additions of the aluminum, titanium and carbon, complex precipitation occur during heat treating in temperature range from approximately 550 to 900 °C [1-8]. Intermetallic phases are Ni_2(Cr,Mo), γ'' (Ni_3Nb) and δ (Ni_3Nb). The γ'' phase is metastable with ordered DO_{22} structure while the δ phase is the stable form with DO_a structure [1,3,4]. In the same temperature range the precipitation of the $M_{23}C_6$ and M_6C secondary carbides was also reported [1,5-9]. The metastable γ'' phase usually forms in a γ matrix in the form of disc shaped or ellipsoidal particles and improves hardness, tensile strength and low cycle fatigue [8,10]. The γ›› phase precipitates in temperature range from approximately 550 to 750 °C [1,3,4,8]. At higher temperature range the precipitation domain of γ›› intersects with the precipitation domain of δ phase [1,3,4,8]. The precipitation domain of δ phase is approximately from 650 to 900 °C. For this alloy the best mechanical properties by thermal aging were achieved at temperature of 650 °C [7,8]. At temperature of 650 °C, the γ›› phase precipitates first, following with precipitation of secondary carbides $M_{23}C_6$ and δ phase. But the precipitation kinetics of these phases is strongly influenced by state of the alloy before thermal aging [4,5,7-9]. In this study thermal aging of Inconel 625 in hot worked state was conducted at temperature of 650 °C with different duration of treatment to study the influence of thermal aging on microstructure evolution and hardness.

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EKSPERIMENTAL

In this work, nickel superalloy Inconel 625 was used with chemical composition given in Table 1. Inconel 625 was obtained in hot rolled state and cooled in air. The samples were cut from the rolled plate and thermally aged at 650 °C for 0,5; 5; 25; 240; 504; 1 000 and 2 000 h. After heat treatment the samples were cooled in water. The samples were prepared and etched with 30 ml lactic acid, 20 ml HCL and 10 ml HNO_3 for optical microscopy (OM) and scanning electron microscopy (SEM). OM was conducted with Nikon Microphot FXA, while SEM with FE-SEM JEOL JSM 6500F field-emission scanning electron microscope. Different phases were identified by morphology, chemical composition and electron backscatter diffraction analysis (EBSD).

Table 1 **Chemical composition of Inconel 625 / wt. %**

Elements	Comp. / wt. %
Ni	Bal.
Cr	21,8
Mo	8,7
Nb	3,5
Ti	0,29
Si	0,24
Fe	0,15
N	0,018
C	0,012

The Vickers hardness measurements were conducted on the sample before and after thermal aging. The force used during hardness measurements was 0,5 N. The hardness measurement of a nitrides and carbides was avoided.

RESULTS AND DISCUSSION

The OM and SEM images prior to heat treatment showed γ-matrix with variable sized crystal grains, high amount of twins and some sharp (Nb,Ti)(C,N) (Figure 1). Under OM and SEM, no other phases were observed before thermal aging. The first sign of precipitation in SEM images was observed in the sample heat treated for 25 h (Figure 2). These were small barely visible particles in γ matrix and irregular precipitates at the grain boundaries (Figure 2). In the sample heat treated for 240 h the precipitates in γ-matrix have grown and have elliptic morphology (Figure 3). Based on morphology this are precipitates of γ'' phase (Figure 2) [2-8]. Compared to sample heat treated for 25 h, the grain boundaries appear to be highlighted due to precipitates with irregular shape (Figure 3).

The grain boundaries of sample heat treated for 504 h visually appear even more highlighted compared to sample heat treated for 240 h, due to precipitates on a grain boundaries (Figure 4). These are $M_{23}C_6$ secondary carbides, based on EBSD Kikuchi patterns (Figure 5) and higher content of chromium and carbon (Table 2) compared to γ-matrix (Figure 6, Table 2). On some grain boundaries the precipitates of $M_{23}C_6$ secondary carbides connected and formed continuous film. While on other grain boundaries, films of secondary carbides are interrupted by thin needless and larger squared particles (Figure 3). The morphology of thin needles and squared particles is similar to δ phase [4,5,8].

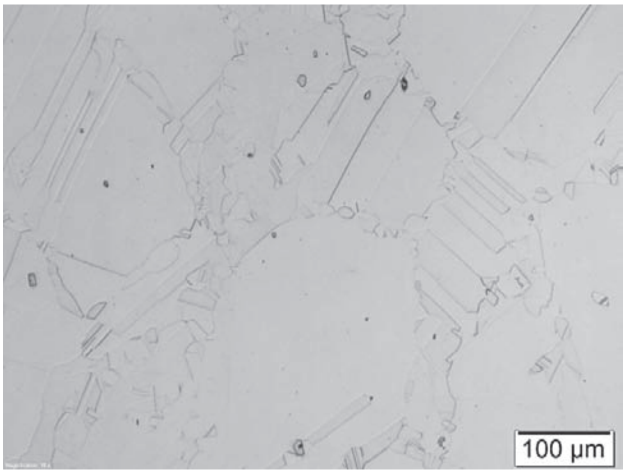

Figure 1 Microstructure of the sample before thermal aging

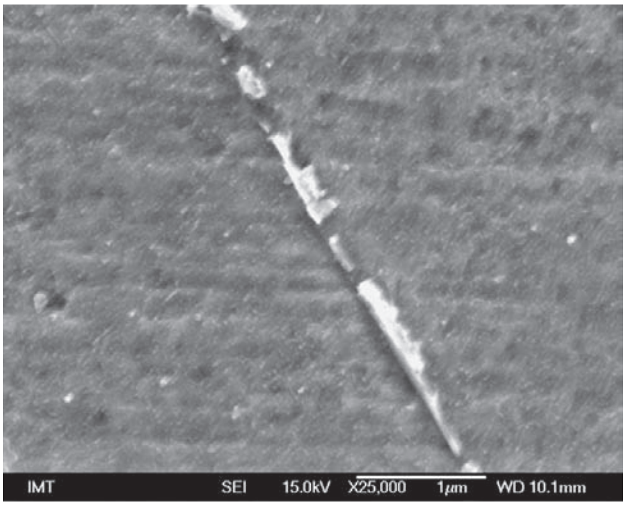

Figure 3 Microstructure of the sample heat treated at 650 °C for 240 h

Large number of twins appeared highlighted due to small precipitates at twin boundaries. The precipitates at the twins boundaries are probably $M_{23}C_6$ secondary carbides and γ'' phase [3,8]. However, the precipitation did not occur on all twin and grain boundaries and some were completely empty. In the SEM image of sample heat treated for 504 h, the γ'' phase precipitates in the γ matrix appear to be homogenously distributed (Figure 4).

With longer duration of heat treatment the number of needles of the δ phase on grain boundaries increased. In sample heat treated for 2 000 h the colonies of δ phase needles were also observed on grain boundaries (Figure 7).

The results of Vickers hardness measurement are shown in Figure 8. The initial state, prior to heat treatment is marked with 0. The initial state had hardness values approximately 277 HV, while samples heat treated for 0,5 and 5 h have hardness values approximately 280 and 283 HV, respectively. The significant increase of hardness was observed in the sample thermally aged

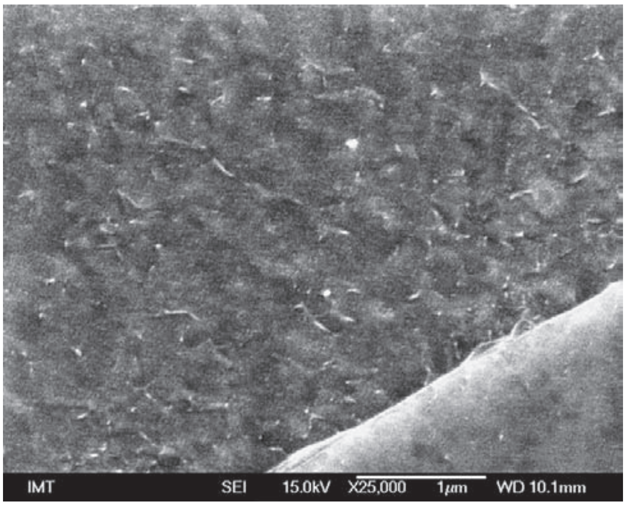

Figure 2 Microstructure of the sample heat treated at 650 °C for 25 h

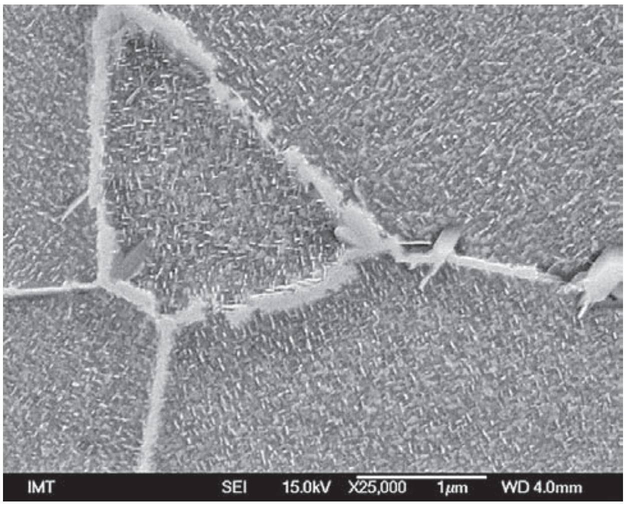

Figure 4 Microstructure of the sample heat treated at 650 °C for 504 h

Table 2 **The composition of γ-matrix and $M_{23}C_6$ secondary carbide in sample heat treated for 504 h**

Phase	γ-matrix / wt. %	$M_{23}C_6$ / wt. %
C	-	8,0
Cr	21,9	25,2
Ni	62,6	52,2
Nb	4,7	3,5
Mo	10,8	10,9

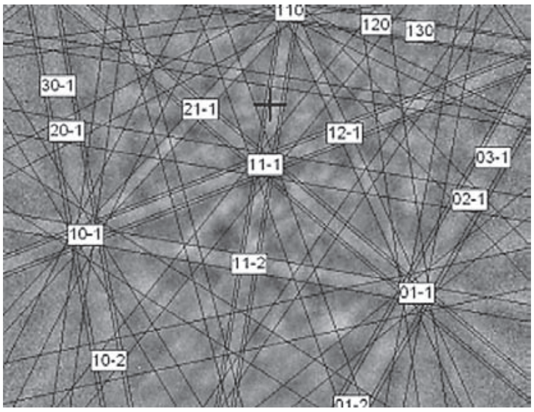

Figure 5 Kikuchi pattern of $M_{23}C_6$ secondary carbide of sample heat treated for at 650 °C 504 h, 10 000x

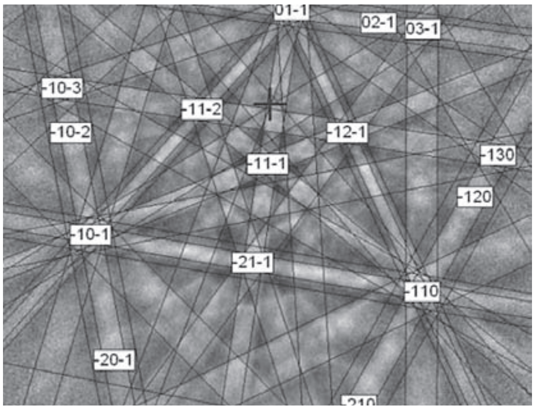

Figure 6 Kikuchi pattern of γ matrix of the sample heat treated at 650 °C for 504 h, 10 000x

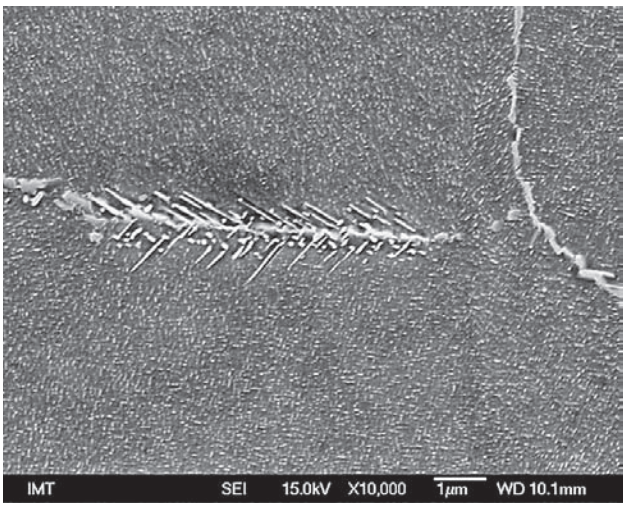

Figure 7 Microstructure of sample heat treated at 650 °C for 2 000 h

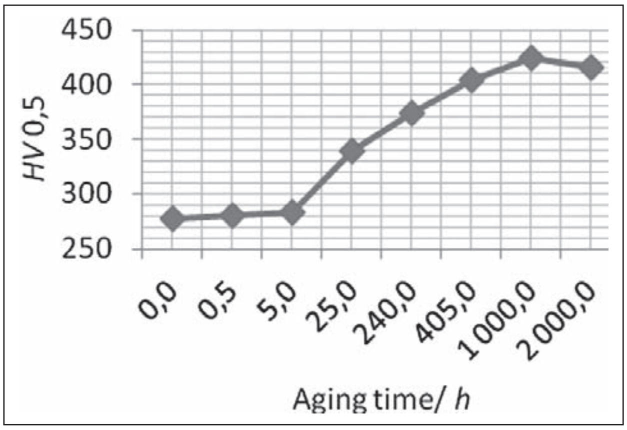

Figure 8 The results of Vickers hardness measurements

for 25 h, approximately 339 HV. The values for hardness increased with duration of thermal aging and reached maximum values in the sample heat treated for 1 000 h approximately 424 HV. With further thermal aging the values for Vickers hardness decreased and reached approximately 415 HV in sample heat treated for 2 000 h.

The increased hardness of samples thermally aged for more than 5 h is attributed to the precipitation of γ'' phase in γ matrix (Figure 2), similar to other reports [2-6,8]. Even though, prior to the thermal aging the alloy was not solution treated, the aging response is strong. Due to further precipitation of the γ'' phase the hardness increased with duration of thermal aging and reached maximum values in samples thermally aged for 1 000 h. The precipitation of δ phase was observed earlier compared to literature [1,6]. Kinetics of the δ phase precipitation changed, possibly due to deformed state after hot rolling, which promoted the precipitation of δ phase [8]. The hardness decreased in the sample thermally aged for 2 000 h, due to δ phase precipitation that grows on behalf of the γ›› phase. Even though the Vickers hardness increased in samples thermally aged from 25 to 1 000 h, the influence of the $M_{23}C_6$ secondary carbides and the δ phase precipitation cannot be completely deduced from results of Vickers hardness measurement alone. Because the $M_{23}C_6$ secondary carbides and δ phase precipitated on grain boundaries in large quantity, other mechanical properties like tensile strength and rupture strength are most likely decreased in samples thermally aged for more than 240 h [10]. For samples thermally aged for 25 and 240 h the tensile strength and rupture strength was most likely improved. But further tests, like tensile, Charpy or creep test should be conducted to verify this.

CONCLUSIONS

In this study thermal aging of Inconel 625 in hot rolled state was conducted at temperature of 650 °C with different treatment duration to study the influence of thermal aging on microstructure evolution and Vickers hardness. From SEM images the γ›› phase and δ phase precipitation was first observed in sample thermally aged for 25 h and 504 h respectively. The hardness increased with precipitation of γ›› phase and reached maximum values of approximately 424 HV in sample thermally aged for 1 000 h even though the resolution treatment was not used before thermal aging. In the sample thermally aged for 2 000 h the hardness decreased due to δ phase precipitation.

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