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Research Note

Aging behavior and microstructure evolution of a cold-drawn Ni-Co superalloy

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Abstract. Age hardening behavior and microstructure of superalloy AEREX 350^{TM} in commercially cold-drawn condition were studied. It was found that aging the cold drawn alloy significantly facilitated the formation of major phases of gamma prime and eta, in AEREX 350^{TM}. Interestingly, the minimum temperature of formation of gamma prime and eta phases in the cold drawn alloy was lower by about 30°C and 5°C, respectively, to that previously reported for the solution treated alloy. Cold working, however, did not considerably influence the maximum temperature of stability of these phases. In addition, Widmanstätten morphology of eta phase significantly improved in the cold drawn samples compared to that seen in the solution treated alloy. Diffusion through dislocation network could have facilitated growth of major phases whereas its influence on dissolution temperature of these phases was negligible. In addition, it was suggested that cold working might increase the density of hcp nuclei in the fcc matrix. This may justify the observed enhancement of the Widmanstätten morphology of eta phase in these samples following the aging process.

1. Introduction

The nickel-cobalt base superalloy AEREX 350 is a member of Multiphase^{TM} (MP) family, a group of thermo-mechanically processed alloys, developed for high-quality applications including aerospace and petrochemical industries. MP alloys have been subject to many investigations upon the past decades [1-6]. AEREX 350, the latest member of this family, has shown superior creep resistance and rupture properties compared to other nickel-based alloys such as Waspalloy, Alloy 718 and Udimet 720 [7]. Previous investigations have shown two main phases in this alloy: (i) major strengthening phase is γ'; an ordered phase with an FCC($L1_2$) structure and a nominal composition of $Ni_3(Al,Ti)$ that increases the strength of the alloy, due to coherency strains, and (ii) η phase with HCP(DO24) structure and Ni_3Ti composition. γ' phase distributes throughout the FCC matrix as fine precipitates while η phase appears with two major morphologies, including the discrete blocky precipitates at grain boundaries that is the predominant form at lower aging temperatures and may enhance high-temperature properties of the alloy. This may be explained by taking into account the difficulty of grain boundary migration under this situation. Widmanstätten ones form along with the (111) crystallographic planes of the matrix at higher aging temperatures and deteriorates the performance of the alloy for room temperature applications requiring strength and ductility. Con-

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trolling the microstructure is therefore a necessary requirement for optimizing the properties of AEREX 350. Important aspects of microstructure of AEREX 350 and the evaluation of cold work influence on the microstructure of this alloy have been investigated in recent studies [8-12]. For instance, precipitation behavior of solution-treated AEREX 350 was investigated [9], or some researchers [12,13] evaluated the influence of cold working prior to aging on the microstructure of the alloy. This research study, thus, aims (i) to characterize the aging response of the cold-drawn alloy with respect to hardness values of the alloy AEREX 350, and (ii) to determine the effects of the cold work prior to aging on the stability of phases in this alloy.

2. Material and experiment

The material used in this investigation was supplied by SPS Technologies, Jenkintown PA, as cold-drawn rods of AEREX 350 (25Co - 17Cr - 3Mo - 2W - 4Ta - 2Ti - 1Al - 1.1Nb - 0.015C - 0.015B - balance Ni in weight percent). AEREX 350 is made by a triple melting route (VIM+ESR+VAR) to ensure an inclusion free material desirable for further thermo-mechanical processing. The cast product was hot rolled to rods, 18 mm in diameter, solution treated for 1.5 hour at 1150°C in argon atmosphere and subsequently water quenched and finally cold drawn to rods, 14 mm in diameter. Three millimeter thick specimens were cut perpendicular to the drawing axis. The samples were aged at temperatures ranging from 660 to 1100°C followed by quenching in water.

The Vickers hardness of aged samples was measured by a Wolpert hardness tester by 30 kgf. At least four indentations were made for each hardness measurement and the average hardness was plotted against aging times. The samples for the microscopy studies were carefully ground and polished. An etching solution of marble etchant, made of 15 g $CuCl_2$, 150 ml methanol and 100 ml HCl, was applied to the surface of the polished samples. These samples were studied using a CARL ZEIS JENA, NEOPHOT 32, optical microscope and a JEOL JXA-840 Scanning Electron Microscope (SEM) to document the changes in the underlying microstructure associated with aging. For a more detailed investigation, a number of samples were studied by Transmission Electron Microscopy (TEM) using a PHILIPS C20 TEG-FEG instrument at 200 kV and two beam condition imaging using a double tilt holder. The final thinning of the TEM samples was done by electropolishing them in a jet polishing unit using a solution of 240 ml methanol, 60 ml H_2SO_4 and 15 ml H_3PO_4 at room temperature with a voltage of 20 V.

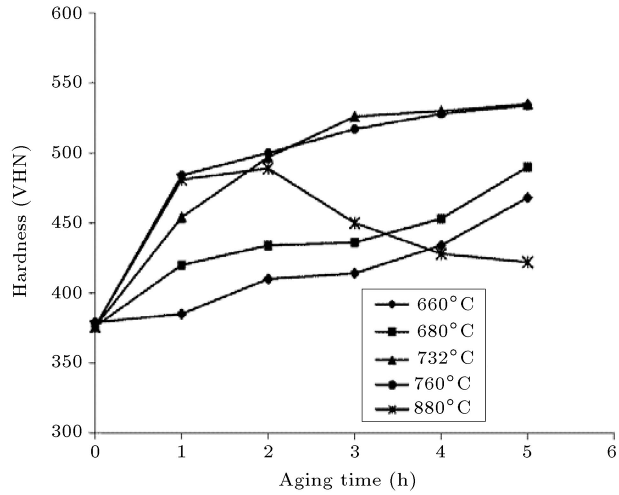

Figure 1. Influence of the aging time on the average hardness of AEREX 350 alloy at aging temperatures 660 to 880°C.

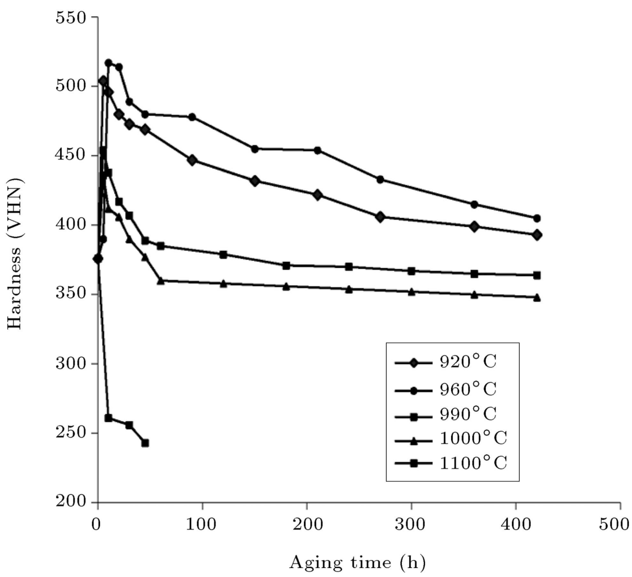

Figure 2. Influence of the aging time on the average hardness of AEREX 350 alloy at aging temperatures 920 to 1100°C.

3. Results

Results of hardness testing are summarized in Figures 1 and 2. As shown, aging at 660°C and 680°C causes a steady but slow increase in the hardness values with increasing the aging time. A previous investigation showed no measurable change in the hardness in the solution-treated samples aged at temperatures up to 680°C [9]. At aging temperatures of 732 and 760°C, however, a significant increase in hardening response occurs. At higher temperatures up to 1000°C, the hardness of cold-drawn AEREX 350 increases initially, reaches a maximum value, beyond which shifts to shorter times with increasing aging temperature, and eventually falls to lower values. For long aging times at

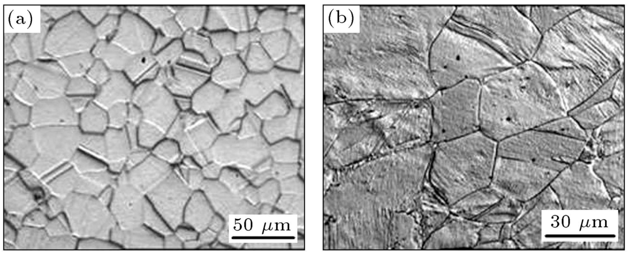

Figure 3. Optical micrographs of AEREX 350 alloy as (a) solution-treated, and (b) cold-drawn.

these temperatures, the final hardness of the material reaches a constant value of about 400 HV30. The rate of this falling in hardness was higher in the temperature range of 990 to 1000°C compared to that measured at 920 and 960°C especially at 880°C. At 1100°C, the material is actually solution treated and reaches to a minimum hardness of 210 HV30 after one hour.

Figure 3(a) and (b) show optical micrographs of solution-treated and cold-drawn AEREX 350 alloy, respectively. Annealing twins (straight bands) observed inside the grains are characteristic features of microstructure in low stacking fault energy alloys. While these bands are straight in the solution-treated samples, they appear as curved bands in the cold-drawn samples. This is due to the inhomogeneous deformation inside the grains that causes the curvature of lattice planes. In addition, fine deformation markings are seen in a number of grains in Figure 3(b). These markings are known to be deformation twins formed during cold work process [11].

Figure 4 shows a TEM bright-field micrograph of γ' precipitates in a sample aged at 880°C for 2 h. These precipitates, scattered uniformly throughout the deformed matrix, have been formed during aging process [8,9]. Figure 5 shows a TEM bright-field micrograph of γ' and η phases in a sample aged at 960°C for 45 min. It appears that growth of η phase

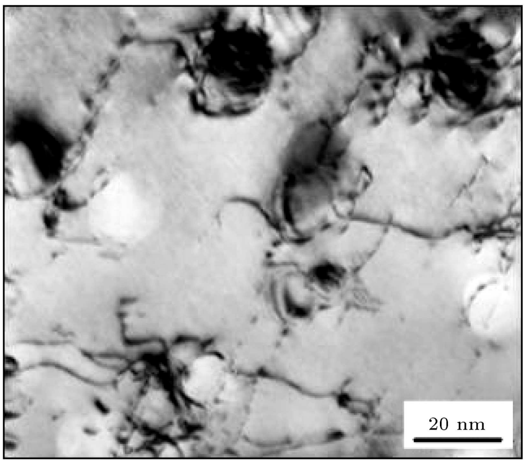

Figure 4. TEM micrograph of γ' precipitates in a sample aged at 880°C for 2 h.

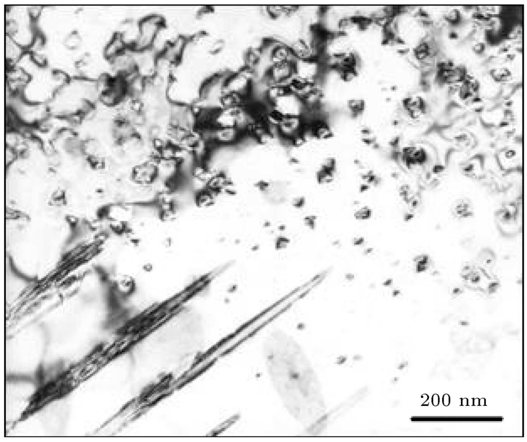

Figure 5. TEM bright-field micrograph of γ' and η precipitates in a sample aged at 960°C for 45 minutes.

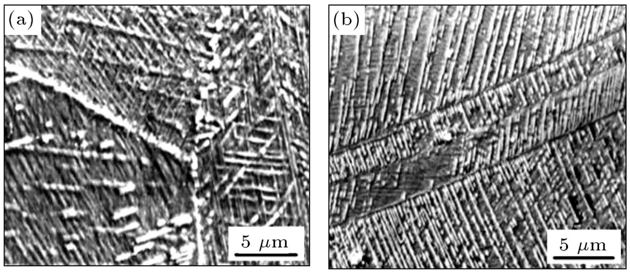

Figure 6. SEM micrographs of a sample aged at 1000°C for 7 h: (a) Grain boundary and Widmanstätten morphologies of η phase; and (b) grain and twin interior morphologies of η phase.

Figure 7. TEM bright-field micrograph of η phase in a sample aged at 1000°C for 1 h. Arrows show two blocky and Widmanstätten morphologies of η phase left behind the recrystallization front.

occurs at the expense of γ' phase as implied by the precipitate free zone formed around η phases [13].

Figure 6(a) and (b) show η phase formed at grain boundary (discrete blocky), grain interior (Widmanstätten morphology) and twin interior in a sample aged for 7 hours at 1000°C. Figure 7 shows a TEM bright-field micrograph of η phase in a sample aged at 1000°C for one hour. Both blocky and Widmanstätten morphology of η are seen behind the recrystallization front. While η phase was stable at this temperature, no γ' precipitate was present in either the recrystallized region or the deformed matrix in this sample. One important aspect of the recrystallization behavior of alloys containing coherent and semi-coherent precipitates is the influence of these phases on recrystallization behavior of the alloy. These precipitates due to the requirement of coherency loss may inhibit the passage of the large angle boundaries involved in recrystallization. The effects of η and γ' precipitates on the recrystallization behavior of AEREX 350 have not been the subject of this work.

4. Discussion

A number of studies [8-11] have shown that the observed increase in the hardness of age-hardened AEREX 350 is due, principally, to the presence of finely dispersed γ' precipitates. In addition, the fall in the hardness after a peak in the aged samples has been attributed to the coarsening and/or dissolution of γ' precipitates with increasing the aging time and temperature [9]. For example, with increasing the aging time at 880°C, γ' precipitates grow and, therefore, the hardness starts to decrease after reaching the maximum extent. At 920 and 960°C, the fall in hardness after reaching a peak value occurs at a lower rate compared to that observed in the samples aged at lower temperatures (880°C), as depicted in Figures 1 and 2. This behavior is concurrent with the precipitation of a large volume fraction of Widmanstätten η. Formation of this morphology is known to increase strength of the material and decrease its ductility [10]. The high rate of the fall in hardness of the samples aged at higher temperatures (990-1100°C) is attributed to the recrystallization of the alloy.

Formation of grain boundary η in cold-drawn AEREX350$^{\text{TM}}$ occurs at lower temperatures while Widmanstätten morphology of η is prevailing at higher temperatures. This is similar to that reported for the solution-treated alloy [9]. At low temperatures where matrix diffusion is slow compared to grain boundary diffusion, grain boundary η grows much faster than Widmanstätten η. With increasing the temperature, however, lattice diffusion becomes dominant and Widmanstätten morphology prevails [9]. Also, it should be noted to the γ'-free zone formed around η precipitate in Figure 5, thereby suggesting that these precipitates grow at the expense of γ' [9,12,14]. This has been attributed to the thermodynamic instability of γ' with respect to the η at high temperatures [9,15-16].

In a previous study [9], the stability of γ' and η phases in the solution-treated samples aged at different aging temperatures was investigated. The ranges of stability of γ' and η was found to be 690-980°C and 800-1055°C, respectively. A detailed TEM investigation was conducted on the samples aged at different temperatures to evaluate the range of stability of these phases in the cold-drawn samples. The diffraction patterns taken from these investigations showed the hexagonal structure of the new phase. From the collected diffraction data and microscope constants, the new precipitate was identified as Eta (η) phase with D024 structure and lattice parameters a and c of 0.49 nm and 0.81 nm, respectively. These values are comparable to the corresponding X-ray data presented in a recent study on the AEREX 350 alloy [17]. Results of these investigations are summarized in Table 1. Interestingly, while the minimum temperature of γ' formation is lowered by about 30°C in the cold-drawn material, the lower limit of η formation was reduced by only 5°C. This may be attributed to the accelerated diffusion through the dislocation network formed during cold working. At low temperatures, diffusion through dislocation pipes may significantly contribute to the total mass transfer while at higher temperatures this contribution becomes less important. Therefore, the effect of cold working on lower formation limit is expected to be more significant for γ' compared to η, because, it forms at lower temperatures. Since, minimum stability temperature of γ' phase is lower than η phase, the effect of cold working on nucleation rate of γ' phase is more than η phase. Thus, in cold-drawn alloy with respect to solution-treated alloy, difference of minimum forming temperature of γ' phase is more than η phase. The upper limits of existence of these phases are unaffected by the application of cold working due to the thermodynamic instability of these phases at the corresponding dissolution temperatures.

Table 1. Proposed range of stability of observed phases in the solution-treated [9] and cold-drawn AEREX 350 alloy.

Phase	Solution-treated [9]		Cold-drawn (this work)	
	Min	Max	Min	Max
γ'	690°C	980°C	660°C	980°C
η	800°C	1055°C	795°C	1055°C

5. Conclusions

Firstly, as shown in previous investigation, no measurable change in hardness of cold-drawn AEREX 350 alloy occurs at aging temperatures below 660°C. But, aging at 660°C and 680°C causes a slow increase in hardness while the hardness of material increases significantly at 732°C and 760°C due to the presence of coherent γ' precipitates. Aging at higher temperatures up to 1000°C causes an initial increase in hardness followed by a fall to lower values after reaching a maximum due to the coarsening of these precipitates and/or recrystallization. At 1100°C, no increase in hardness is observed indicating that this temperature is above the limit of stability of all precipitates in AEREX 350.

Secondly, the ranges of stability of γ' and η phases in cold-drawn AEREX 350 are found to be 660-980°C and 795-1055°C, that minimum temperatures of formation of these phases is about 30°C and 5°C lower than that of the solution-treated alloy, respectively.

Thirdly, the larger volume fraction of Widmanstätten η phase formed in the cold-drawn samples compared to the solution-treated alloy, is probably related to the larger number of hcp nuclei formed during pre-aging cold work. The much slower fall in hardness at 920 to 960°C compared to those observed at lower temperatures is related to the extensive formation of high strength Widmanstätten η precipitates.

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Biographies

Hasan Ghiasi received his BSc degree in Metallurgical Engineering from Sharif University of Technology in 1998, and his MSc degree from Tehran University in 2000. Since then, he has been working as a faculty member in laboratories, research and industrial projects, in the Institute for Technology Development. He has accomplished 15 research and technical projects and published 16 papers and 2 books.

Sirous Asgari received his BSc degree in Metallurgical Engineering in 1985, and his MSc degree in 1987 both from Tehran University. He worked as a faculty member during 1999-2001 at the Department of Metallurgical and Materials Engineering, Tehran University, and since then, at the Department of Materials Science and Engineering, Sharif University of Technology. He is interested in the fields of processing and characterization of new materials for Li-ion battery applications, processing and characterization of colloidal quantum dots, surface treatment of Ti alloys for implant applications, micro-mechanisms of strain hardening in FCC alloys, and structure-property correlation in advanced metallic systems.