Analysis of the structure and precipitation strengthening in a creep resisting Fe–Ni alloy

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Materials

ABSTRACT

Purpose: This study describes relationships between the kinetics of precipitation and growth of the intermetallic phase γ' [$Ni_3(Al,Ti)$] and the strengthening magnitude obtained in a high-temperature Fe–Ni alloy of the A-286 type. In order to accomplish the goal of the study, the author used the LSW coagulation theory and Brown and Ham's conventional analysis of strengthening by ordered particles.

Design/methodology/approach: The samples were subjected to a solution heat treatment at 980°C/2h/water and then aged at 715, 750, and 780°C, with holding times 0.5-500h. The heat-treated samples were subjected to structural analyses (TEM, X-ray diffraction) and analyses of mechanical properties (hardness test, static tensile test, and impact test).

Findings: Based on direct measurements on the TEM pictures converted into binary images, the basic parameters of the γ' phase, i.e. average diameter, volume fraction, and the average distance between particles, were determined. Linear regressions between the average particle diameter of the γ' phase and the aging time ($t^{1/3}$) were conducted for the analyzed aging temperatures on the basis of the LSW theory. The author carried out analyses of strengthening and flow stress ($\Delta\tau$) increases as a function of the particle size of the γ' phase and determined the value of the antiphase boundary energy (γ_{APB}) for the analyzed Fe–Ni alloy.

Practical implications: The obtained relationships of the growth of the γ' phase particles as a function of temperature and aging time can be used to determine the magnitude of strengthening and flow stress in high-temperature Fe–Ni alloys during extended aging or usage.

Originality/value: This study exploits LSW coagulation theory and Brown and Ham's conventional analysis to describe precipitation strengthening by ordered particles of the intermetallic phase γ' in a high temperature Fe–Ni alloy.

Keywords: Metallic alloys; Heat treatment; Structure; Precipitation strengthening

1. Introduction

Austenitic Fe-Ni high-temperature alloys precipitation strengthened by intermetallic phases display numerous characteristic properties, i.e. [1-5]: excellent mechanical properties, considerable creep resistance and heat resistance at high temperatures, excellent corrosion resistance, high ductility at low temperatures, and are non-magnetic.

The primary strengthening phase, which is precipitated in high temperature Fe–Ni alloys, is the intermetallic phase γ'[$Ni_3(Al,Ti)$]. The process of growth of the phase γ' particles for coagulation controlled with volume diffusion can be described using LSW theory [6,7]. Application of Brown and Ham's [8]

conventional analysis in describing precipitation strengthening enables the determination of the impact of the γ' phase precipitates on the value of critical shear stress or yield point of the aged Fe–Ni alloys. Strengthening by ordered particles of the γ' type is associated with the occurrence of antiphase boundary (APB) on the slip plane of a particle cut by dislocation.

This study applied LSW coagulation theory and Brown and Ham's conventional analysis to describe the precipitation strengthening by particles of the γ' phase in a creep resistant Fe–Ni alloy of the A-286 type during heat treatment.

2. Material and procedure

The examinations were performed on rolled bars, 16 mm in diameter, of an austenitic Fe–Ni alloy. The base chemical composition of the material is given in Table 1.

Table 1.
Chemical composition of the investigated alloy

Content of an element [wt.%]										
C	Si	Mn	Cr	Ni	Mo	V	Ti	Al	B	N
0.05	0.55	1.25	14.3	24.5	1.3	0.41	1.88	0.16	0.007	0.0062

The samples for testing cut off these rods were subjected to solution heat treatment at 980°C for 2 h and water quenched, and then to aging at temperatures 715, 750 and 780°C at holding time from 0.5 to 500 h.

Observations of the alloy substructure were carried out using the thin foil technique on a Jeol JEM-2000 FX transmission electron microscope (TEM). The X-ray phase analysis of isolates was carried out with a Philips PW-1140 X-ray diffractometer.

A quantitative analysis of secondary γ' phase particles, which phase precipitated in the alloy during ageing, was performed on TEM images in bright field mode. Based on direct counting, basic stereological parameters of the γ' phase were determined from binary images, i.e.: the mean diameter ($\overline{D}$), the volume fraction particles (V_V) and the mean distance between particles (l_d). The detailed methodology to obtain these parameters was described in the earlier work [9].

The analysis of the γ' phase growth step was made on the basis of the LSW theory for coagulation on volume diffusion controll according to the relationship provided by Kusabiraki *et al.* [10]:

$$\overline{d}^3 - \overline{d}_0^3 = \frac{64\sigma D C_e V_m^2}{9RT} = K't \tag{1}$$

where:
$\overline{d}$, $\overline{d}_0$ - average diameter of precipitates at time,
t, t = 0 - respectively,
σ - interfacial energy between precipitates and matrix,
D - diffusion coefficient of the solute atom in the matrix,
C_e - concentration of solute atoms in the matrix in equilibrium with a particle of an infinite size,
V_m - molar volume of precipitation phase,
R   - gas constant,

T - absolute temperature,
K' - growth rate constant.

Flow stress increments (Δτ) for aged Fe–Ni alloy were plotted as a function of square root of γ' particle size in accord with the Brown and Ham [8,11-15] conventional analysis for ordered-particle strengthening:

$$\Delta\tau = \frac{1}{2b} \cdot \left(\frac{df}{T}\right)^{1/2} \cdot (\gamma_{APB})^{3/2} - \frac{f}{2b}(\gamma_{APB}) \tag{2}$$

where:
d   - particle diameter,
f   - particle volume fraction,
b   - matrix Burgers vector,
T   - dislocation line tension,
γ_{APB} - anti-phase boundary (APB) energy.

3. Experimental results

Application of 1 × step ageing at 715, 750, and 780°C after heat solution treatment and holding times ranging from 0.5 to 500 h results in the processes of precipitation of intermetallic phases of the γ'[Ni₃(Al,Ti)], η[Ni₃Ti], G[Ni₁₆Ti₆Si₇], β[NiTi], and σ[Cr₀.₄₆Mo₀.₄₀Si₀.₁₄] types and of M₂₃C₆ carbide in the Fe-Ni alloy [9]. The intermetallic γ' phase was the primary phase precipitated during alloy ageing. Intensive precipitation of dispersive and spheroidal γ' phase particles characterized by strong cohesion with austenite was observed in the alloy matrix at longer ageing times at 715°C (Fig. 1).
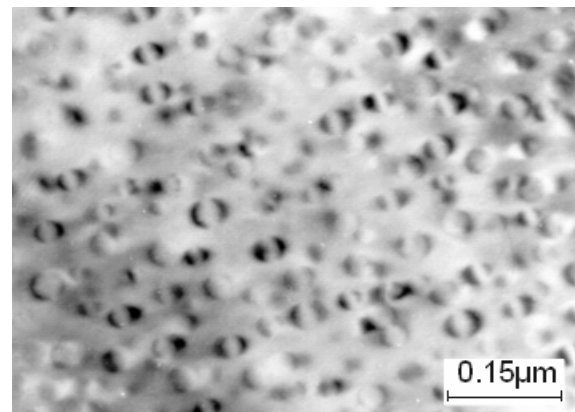

Fig. 1. Structure of the alloy after solution heat treatment and aging at 715°C/150h. Coherent spheroidal γ' phase precipitates in the matrix

Increas in the aging temperature to 750°C intensified the diffusive processes of growth and coagulation of the γ' phase particles, which still retain a spheroidal shape and cohesion with austenite (Fig. 2). At the highest ageing temperature of 780°C, a clear effect of overageing, associated with coagulation of the γ' phase particles, loss of cohesion with austenite and occurrences ofarrangement errors inside the precipitates were observed in the alloy structure (Fig. 3).
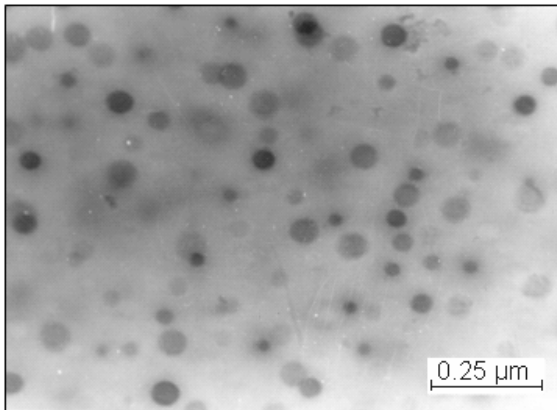
Fig. 2. Structure of the alloy after solution heat treatment and aging at 750°C/300h. Divesified size of spheroidal γ' phase particles in the matrix
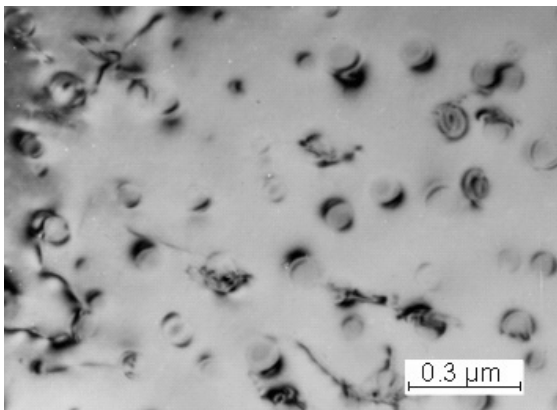

Fig. 3. Structure of the alloy after solution heat treatment and aging at 780°C/500h. Coagulated spheroidal γ' phase particles in the matrix

The kinetics of growth and coagulation of the γ' phase was analyzed based on LSW theory according to equation (1). The relationship between the mean diameter ($\overline{D}$) of the γ' phase particles and ageing time raised to the one third power ($t^{1/3}$) at the aging temperatures of 715, 750, and 780°C is presented in Fig. 4. Linear regressions were obtained for all analyzed temperatures, which demonstrates that coagulation of the γ' phase particles occurs according to LSW theory and is controlled by volume diffusion.

Precipitation strengthening of the Fe–Ni alloy during ageing was described based on results from the structural analysis and investigation of mechanical properties. The initial analysis included plotting graphs of increases in hardness (ΔHV) and yield strength (ΔP.S.) as a function of change in the γ' phase particle size. The obtained increases in hardness (ΔHV) and yield strength (ΔP.S.) as a function of the mean diameter ($\overline{D}$) of the γ' phase particles are presented in Figs. 5,6. It is evident from the plotted curves that the level of strengthening obtained during ageing was highly correlated with the average diameter of the γ' precipitates and the ageing temperature.
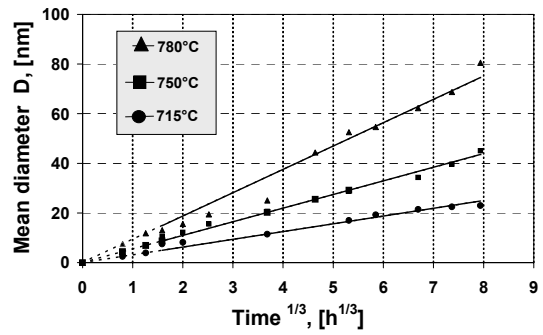

Fig. 4. Relationship between the mean diameter of γ' phase particles and the cube root of aging time at temperatures of 715, 750 and 780°C
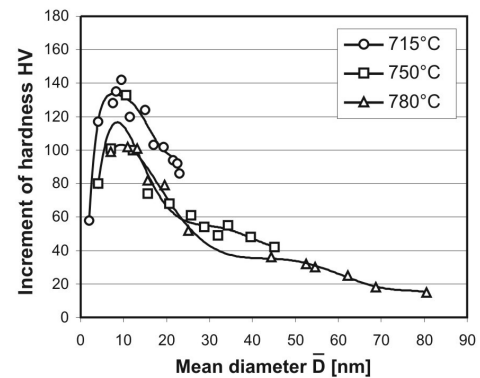

Fig. 5. Relations between increased hardness and mean diameter of γ' precipitates
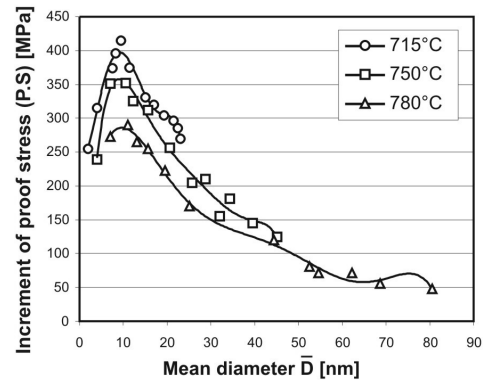

Fig. 6. Relations between increased proof stress and mean diameter of γ' precipitates

The increase in flow stress (Δτ) for the tested alloy was analyzed according to equation (2). In order to determine the values of flow stress for the tested alloy, it was necessary to solve for Δτ from the equation [8,13]: Δτ = σ_a/3 (where σ_a – increase in alloy yield point during aging, M–Taylor's index equals 3.06 for FCC structures).

The resultant values of the flow stress increase (Δτ) as a function of the mean diameter ($\overline{D}$) from the γ' phase precipitates that were presented in graphic form in Fig.7. It is evident from these relationships that the highest increases in flow stress (Δτ = 129-136 MPa) were observed for the ageing temperature of 715°C and the

heated equivalent diameter ($\overline{D}$) of the γ' phase particles of ca. 8-10 nm. Further increase in the particle diameters from 11 to 23 resulted in a rapid decrease in the strengthening level during ageing. For a higher ageing temperature (750 and 780°C), the greatest increases in flow stress were obtained for the particle sizes of $\overline{D}$ = 7-11 nm.
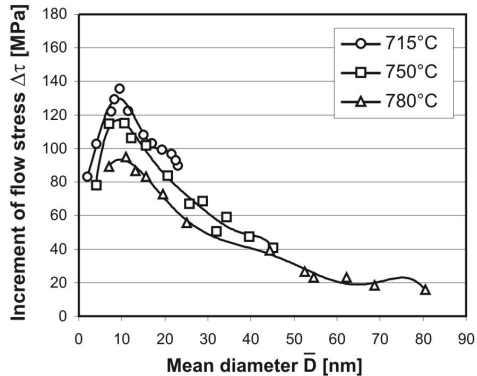

Fig. 7. Relations between increased flow stress and mean diameter of γ' precipitates

Based on the results, it can be concluded that the greatest precipitation strengthening in the tested Fe–Ni alloy occurs at an ageing temperature of 715°C. A detailed graph of the alloy's flow stress increase (Δτ) as a function of the square root $(\overline{D})^{1/2}$ of the particle size was prepared for this temperature (Fig. 8).
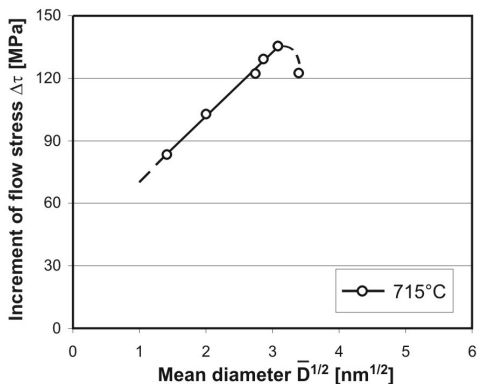

Fig. 8. Flow stress increment due to aging at 715°C as a function of square root of mean diameter of γ' precipitates

It is evident from the figure that from short ageing times to the maximal strength of the alloy, the relationship between Δτ and the particle diameters $\overline{D}^{1/2}$ was linear. The regression slope can be used in equation (2) to determine the APB energy. For the analyzed case, the APB energy in the tested Fe–Ni alloy equalled ca. 210 mJ/m^2. This value is similar to the value of 225mJ/m^2, which was obtained by Thompson and Brooks [13] for a comparable Fe–Ni alloy of the A 286 type.

4. Summary

The intermetallic γ' phase was the primary phase precipitated during the ageing at 715, 750, 780°C and holding time 0.5-500h of the tested Fe–Ni alloy. It was concluded that the mean diameter ($\overline{D}$) of the γ' phase precipitates increases as a function of the cube root of the aging time ($t^{1/3}$), which is in conformity with predictions based on LSW theory.

It was observed that the level of strengthening obtained in the tested alloy significantly correlated with the γ' phase particle size and ageing temperature. The greatest strengthening of the alloy occurred in initial stages of aging for a mean diameter ($\overline{D}$) of the γ' phase particles ranging from 8 to 15 nm.

The analysis of flow stress increase (Δτ) as a function of the square root of the particle size $(\overline{D})^{1/2}$, which was carried out for the ageing temperature of 715°C, yielded a linear relationship to the peak of strength. The APB energy was determined by applying the conventional strengthening theory of Brown and Ham to the line regression slope; its value was estimated to be 210 mJ/m^2.

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