



Coarsening process and precipitation hardening in Fe₂AlV-strengthened ferritic Fe₇₆Al₁₂V₁₂ alloy

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ARTICLE INFO

Keywords:

Ferrous alloy
Age hardening
Coarsening
Electron microscopy
Hardness measurement

ABSTRACT

Strengthening through a homogeneous distribution of a nano-sized second phase is a concept that is proposed to reinforce solid-solution body centered-cubic iron for high-temperature application in fossil-energy power plants. It was shown that these microstructures can be obtained in the Fe-Al-V system with L₂₁-ordered Fe₂AlV precipitates in a ferritic matrix. The effect of aging in the range 600–700 °C on the ferritic Fe₇₆Al₁₂V₁₂ alloy was investigated using Vickers micro-hardness test and transmission electron microscopy. The diffusion screening coarsening theory is used to analyze the ripening kinetics. When volume fraction and mobility of the components in the ternary alloy are considered, the interfacial energy between the matrix and the precipitate was determined as $(18 \pm 3) \times 10^{-3} \text{ J/m}^2$ at 700 °C but increases strongly when the temperature decreases. A classic precipitation hardening behavior has been observed along the time for each aging treatment. At room temperature, the increment of flow stress has a peak of about 450 MPa for a precipitate radius of 10 nm. Quantitative agreement is found with strength values predicted from order strengthening theory, predicting that strength is controlled by a precipitate shearing mechanism for sizes around that of peak strengthening, and the Orowan dislocation bypass mechanism for larger sizes. The APB energy of Fe₂AlV precipitate was estimated to be $(27 \pm 4) \times 10^{-2} \text{ J/m}^2$.

1. Introduction

Ferritic steels are an attractive alternative to austenitic steels for high-temperature applications (e.g., in thermal power plants) due to their lower cost and thermal expansion, and their higher thermal conductivity. The current creep-resistant ferritic steels used in power plants are strengthened by carbides. The creep resistance of those materials decreases in long-term creep tests at temperature over 600 °C, due to the coarsening of the incoherent strengthening carbides [1]. Considerable effort has therefore been made to increase the high-temperature strength of ferritic materials in order to achieve higher operational temperatures and thereby a higher level of efficiency. The approaches that have been followed include a second phase precipitate strengthening. In this respect, the most studied alloy system is the ternary Fe-Al-Ni where there is a miscibility gap between the disordered A2 and the B2-ordered NiAl phases [2,3]; both phases have cubic crystal structure and the mismatch between their lattice para-

eters is quite small which means that NiAl can precipitate coherently within α -Fe. These NiAl-strengthened ferritic alloys have typical chemical composition based on the pseudobinary section Fe+2.5 at% Al-NiAl+1.25 at% Al with a maximum solvus temperature of 1000 °C that decreases with decreasing volume fraction of the B2 phase [4]. It is known from a long time ago that the NiAl precipitates are effective in increasing the strength of an iron (bcc) solid-solution from room temperature up to 600 °C [5], however, the mechanism of hardening by the NiAl precipitates was quantitatively understood in the recent past [6]. At room temperature the coherent NiAl precipitates were found to be sheared by the $\langle 111 \rangle$ slip, which is the primary slip for the bcc matrix, and this shear slip of the precipitates was still active even at 550 °C. Since the creep resistance is one of the most important properties for high temperature applications, the previous research on the Fe-Al-Ni alloy system has also focused on the coarsening behavior of NiAl-type precipitates at high temperatures [7,8] and its relationship with the creep behavior [4,9,10]. Based on these findings,

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<http://dx.doi.org/10.1016/j.msea.2016.12.074>

Received 14 October 2016; Received in revised form 16 December 2016; Accepted 17 December 2016

Available online 19 December 2016

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several works have studied the effect of minor alloying elements in order to improve the aforementioned properties of the NiAl-strengthened ferritic steels [4,7,11,12].

Such a miscibility gap also exists in the Fe–Al–V system between the disordered A2 and L₂₁-ordered Fe₂AlV phases but Fe₂AlV-strengthened ferritic alloys has gained less attention up to now. From the pioneering research of Zhao et al. [13] and Maebashi et al. [14] it is known that this miscibility gap extend roughly in the composition range 5–25 at% Al and 0–25 at% V; it shrinks when the temperature increases up to 750 °C and then a two-phase equilibrium (B2-ordered Fe(Al,V) + L₂₁) appears. In a former work [15] we modeled the bcc phase diagram of the Fe–Al–V system by using ab-initio thermodynamics. It was concluded that this ternary system display two kinds of phase separations of the bcc phase, (A2 + L₂₁) and (B2 + L₂₁), separate by a tie-line. As the temperature increases this tie-line shrinks and moves toward the Fe–V binary system while its direction remains almost parallel to the line from pure iron to the compound Fe₂AlV. So, any vertical section of the ternary diagram parallel to the section containing the line Fe_{1-2x}Al_xV_x will show the border tie-line parallel to the composition axis. Going from aluminum-rich sections to that of the same composition in aluminum and vanadium, the temperature of the border tie-line increases and the two-phase (A2 + L₂₁) coherent microstructures could be generated at higher temperatures and with less aluminum content. In order to preserve the idea of designing a Fe₂AlV-strengthened ferritic alloy with good ductility at low temperature, we investigated the Fe_{1-2x}Al_xV_x isopleth with experiments [16]. Fig. 1 shows the experimentally assessed isopleth, phase separation into disordered A2 and ordered L₂₁ phases occurs for $0.10 < x < 0.15$ at lower temperatures than 720 °C. Above this temperature and with increasing x in the same range of composition may appear the following regions: (A2+B2), B2 and (B2+L₂₁). The alloys quenched from 1100 °C and aged at 700 °C/2 h show spherical precipitates of L₂₁ phase for $x \leq 0.13$, for larger values of x precipitate coagulation and coalescence is observed [17]. The hardness values, measured at room temperature, for the alloys with $x \leq 0.13$ and ageing temperature between 650 and 700 °C are comparable to those obtained in the alloy Fe₈₇Al₁₀Ni₃ [7]. Recently, the dependence of yield strength with temperature of Fe₇₂Al₁₈V₁₀ alloy in the as-cast condition and after heat treatment 700 °C/1000 h has been investigated by Senčková et al. [18] with compression test. The flow stress of this alloy, in both heat treated conditions, is even high up to 700 °C and comparable to those observed for the Fe–Al–Ni alloys with coherent microstructures. These authors have also investigated the creep strength of this alloy between 650 and 750 °C in the aforementioned heat treated conditions. The dependence of the secondary creep rate with the applied stress measured at 700 °C predicts a steady-state-creep rate of approximately 10^{-10} s^{-1} at a stress level of 35 MPa which is less but

close to the tolerable creep strain rate under operation conditions in fossil-energy power plants [1].

This study has been planned to provide basic information for designing Fe₂AlV-strengthened ferritic alloys for high temperature applications. On this basis, a ferritic Fe₇₆Al₁₂V₁₂ alloy was investigated. This alloy composition was chosen to optimize the precipitate volume fraction as well as to avoid precipitate coagulation and coalescence. The coarsening kinetics of Fe₂AlV-type precipitates is examined by the ripening model in multicomponent alloys and its controlling factors of interfacial energy, diffusivities, and alloying element partitioning are discussed. Finally, the increment of strength at room temperature caused by the precipitates was evaluated by precipitation strengthening theory, and the predictions were compared with the experimental data obtained with Vickers micro-hardness test.

2. Experimental procedure

The alloy ingot was melted in an electric arc furnace with tungsten electrode and water cooled copper crucible under argon atmosphere. The purity of the used components was 99.97% Fe, 99.99% Al and 99.7% V. The ingot was then heated in an electric furnace under argon atmosphere up to 1100 °C and hot rolled in several passes ($\epsilon < 0.1$) to form a plate with about 3 mm thickness. The average chemical composition of the alloy plate was measured by electron-probe microanalysis in Cameca SX50 equipment. The value was obtained from the average and standard deviation of 50 measurement points along a straight line of 10 mm. Finally, the plate was fractionated by electrodischarge-machining (EDM) in two types of samples: Ø3×3 mm disc shape and 6×8×3 mm cuboid shape.

All the phase transformation temperatures were determined from differential scanning calorimetric (DSC; Setaram LABSYS evo) measurements with heating rate of 5 °C/min under an argon flow of 25 mL/min. Experiments were carried out with a disc shape sample in alumina crucible. The measurement was made during a heating run after the following conditioning cycle: heating to 1100 °C, hold for 900 s, cooling to 600 °C and hold for 600 s.

The short aging treatments (less than 10 h) were performed on the disc shaped samples using the DSC, while for longer aging time we used the cuboid samples and an electric resistance furnace without inert atmosphere. All aging treatments started after a solution heat treatment 1100 °C /900 s with subsequent cooling to the aging temperature.

The microstructures formed by isothermal ageing were investigated by means of a Transmission Electron Microscope (TEM) (Philips CM200 operated at 160 kV) and the chemical composition of the precipitate phase was analyzed by in-situ Energy Dispersive Spectroscopy (EDS-TEM) (EDAX Apollo X – Philips CM200). To

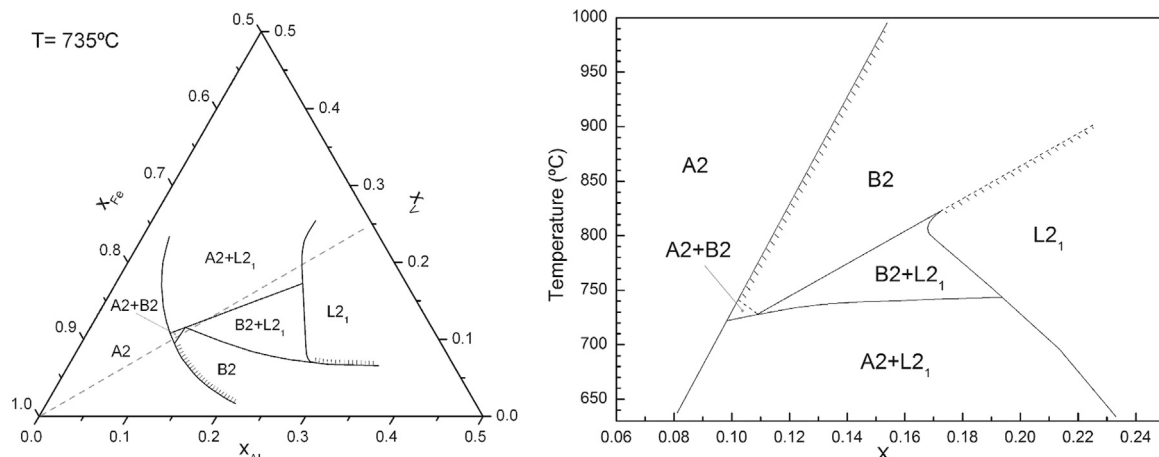


Fig. 1. The experimentally assessed Fe_{1-2x}Al_xV_x isopleth in the Fe rich corner of the Fe–Al–V ternary diagram [16].

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