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Evolution of the microstructure and magnetic properties of as-cast and melt spun Fe_2NiAl alloy during aging

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ABSTRACT

Fe_2NiAl-based alloy with the nominal composition $Fe_{51.1}Ni_{23.5}Al_{23.7}Si_{1.7}$ was prepared by casting and melt-spinning. Comparison of the phase composition, microstructure and magnetic properties of water-quenched bulk samples and melt spun ribbons after isothermal aging in the 500–900 °C range were carried out. TEM investigations of the decomposition of the solid solution into β- and β_2 phases during cooling or quenching and subsequent aging have revealed different types of decomposition products. The optimal periodic modulated structure with coercive force $H_c \sim 700$ Oe was observed after cooling of as-cast alloy at a critical rate. In this structure the paramagnetic β_2 phase forms a continuous network that isolates elongated single domain ferromagnetic β particles. The water-quenched bulk samples and melt spun ribbons were characterized by zone structure with zones about 10 nm and 4 nm in size. The isothermal aging of quenched samples resulted in the formation of modulated microstructure dissimilar to those of the optimal state. The coarsening of ferromagnetic β particles as well as deterioration of the magnetic insulation of β particles occur in bulk samples after aging at $T_{ag} > 700$ °C that decreases $H_c \le 350$ Oe. The dependence $\delta_M(H)$ was measured and negative values of $\delta_M(H)$ in the $H = 0$–2000 Oe range indicate that magnetostatic interactions between the β particles are dominant. The melt spun ribbons were characterized by the presence of antiphase domain boundaries (APD) and discontinuous precipitation (DP) products at grain boundaries (GB). The cellular areas at GBs consisting of alternating lamellas of β′- and β_2' type phases were formed after aging the ribbons at $T_{ag} > 500$ °C. At $T_{ag} > 700$ °C the modulated structure formed inside grains and the wide intergranular double-layer of β and β_2 phases develops by the coalescence of the primary DP products that decrease $H_c \le 250$ Oe. MFM image of the magnetic structure correlated with the microstructure of the ribbon. The coarse domains were observed in the wide intergranular layer of β phase. The fine scale domains observed inside the grains indicates the magnetic domains formed as a result of magnetic interaction between the β particles.

1. Introduction

The rare-earth supply crisis in the last decade has stimulated an active search for new hard magnetic materials as the alternative for rare-earth permanent magnets. The other approach of recent investigations consists in the improvement of the properties of traditional non-rare-earth permanent magnets, including Alni and Alnico alloys. These magnets deserve the special focus because they exhibit good magnetic characteristics, which are essential for permanent magnets used in electric vehicles. Being known since the 1931s [1], the Alni magnets were improved later mainly by empirically optimized processing [2–9]. The coercive force (H_c) of this class of magnets gains by the spinodal decomposition (SD) of the solid solution into magnetic Fe,Co-rich precipitates (β phase) within nonmagnetic NiAl-rich matrix (β_2 phase); H_c increases with increasing the magnetization difference between β and β_2 phases as well as the shape anisotropy in the ferromagnetic β phase. The spinodal decomposition leading to the formation of periodic modulated microstructure takes place during cooling at a critical rate from the high temperature single-phase region below the solubility curve (heat treatment HT-I) or during aging of preliminary homogenized and quenched in water (HT-II) Alni alloys [2,3]. The aging of Alnico-type alloys, which is performed in a magnetic field, gives rise to the formation of modulated microstructure, in which the β phase precipitates are elongated mainly along the applied field direction [4,5]. Recent investigations of Alni [10–12] and Alnico [13–15] using modern characterization tools

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gave new results about nanostructures of spinodal transformation products, chemical composition of β and β_2 phases, anisotropy due to shapes and dimensions, magnetic domain structure and domain wall pinning mechanism. These results allow one to suggest that Alni-based alloys could be further improved [16,17] and may serve as potential candidates for replacing Nd-based magnets in vehicle engines. The preparation of Alni and Alnico alloys in the form of nano-crystalline or nano-structured materials stimulates investigations in this field because these structures exhibit the properties distinct from those of bulk alloys. In the last decade, few studies reported the influence of the confined geometry on spinodal decomposition in Alnico [18,19]. Thus, the effect of cooling rate on the SD structure in Alni and Alnico melt-spun alloys was observed and the coercivity was found to depend strongly on sizes of spinodal structures [11,12,20]. However, the study of spinodal transformations and its corresponding influence on the microstructure and magnetic properties in water quenched bulk samples as well as in melt spun ribbons during subsequent aging has not been carried out. Also, the knowledge of the domain structure in relation to the microstructure is indispensable for understanding the magnetization mechanism in Alni-based alloys. Nevertheless, the observation of the magnetic domain structure in Alni alloys has not been performed.

The aim of this work is to investigate and to compare the evolution of the microstructure of bulk and melt spun Alni alloy during isothermal aging at temperatures in the range of 500–900 °C. The second subject is to investigate the dependence of coercive force on the nanostructured state developed during SD and its corresponding dependence on the spinodal-transformation product structures aged at different temperatures. Also in this work, the topographic microstructure of the Alni ribbon surface was imaged by atomic force microscopy and the magnetic force microscopy was applied for the magnetic domain structure visualization.

2. Materials and methods

The Alni alloy of the nominal composition $Fe_{51.1}Ni_{23.5}Al_{23.7}Si_{1.7}$ was studied. The as-cast alloy was prepared by melting in an induction furnace under an argon atmosphere using pure metals. The melt was cast into a copper finger-mold. The ingot was melted and quenched by melt-spinning with a copper-wheel speed $V\sim40$ m/s. The thickness of the melt spun ribbons was 20–40 µm. Two types of heat treatment were used for as-cast samples. HT-I consists of cooling from 1240 °C (after 20-min holding at this temperature) at a critical rate ($V_{cr}\sim2$ K/min) to room temperature. HT-II consists of quenching in water from 1240 °C, subsequent aging at 500–900 °C for 10 min and quenching in water after holding. The melt spun ribbons were aged in argon atmosphere at 500–900 °C for 10 min and cooled in cold zone of the furnace. The structures of the samples were examined by TEM using a JEM-1400 microscope operated at 120 kV. The thin foils for TEM were prepared by electropolishing at a temperature of −20 °C and a voltage of 23 V using $HClO_4$–ethanol–2-Butoxyethanol (A2) electrolyte and a Struers TenuPol 5 double-jet polisher. As a result of different electrochemical dissolution of β (Fe-rich) and β_2 (NiAl-rich) phases, a microrelief of the ribbon corresponded to the fine structure was generated after the ectropolishing. The ribbon microrelief was studied by atomic force microscopy (AFM) of AIST-NT manufacturer in a taping mode. Magnetic force microscope (MFM) was employed to image the magnetic domain structure in the ribbons with a typical lift height of 50 nm and a high moment Co-coated tip magnetized normally to the sample surface. Magnetic properties of the as-cast samples and ribbons were measured using a PPMS EverCool-II (Quantum Design) magnetometer and a hysteresisgraph AMT-4.

3. Results and discussion

3.1. Structure of as-cast AlNi alloy subjected to cooling from 1240 °C at a critical rate or water quenching and subsequent aging

It is known that the H_c value reached after H-I is higher than that reached after HT-II by a factor of ~1.5 [3], but structural difference after these HT is still not fully understood. Fig. 1 shows (a) bright- and (b) dark-field images of the Alni alloy cooled from 1240 °C at a critical rate. Inset in Fig. 1a shows the electron-diffraction pattern with zone axis [101].

The comparison of bright- and dark-field images in Fig. 1 shows that the sample cooled at a critical rate is characterized by formed periodic modulated structure consisting of elongated β phase particles that are oriented mainly along <100> directions and separated by dark matrix β_2 phase areas. The electron diffraction pattern represented in Fig 1a, inset, is a superposition of two cubic structures, namely the disordered A2 (β phase, Fe-based solid solution) and ordered B2 (β_2 NiAl-based phase) structures; the 010 superlattice reflections correspond to the β_2 phase alone.

As it is seen in the dark-field image of the microstructure, which was taken in image-forming 010 reflection of the B2 phase,

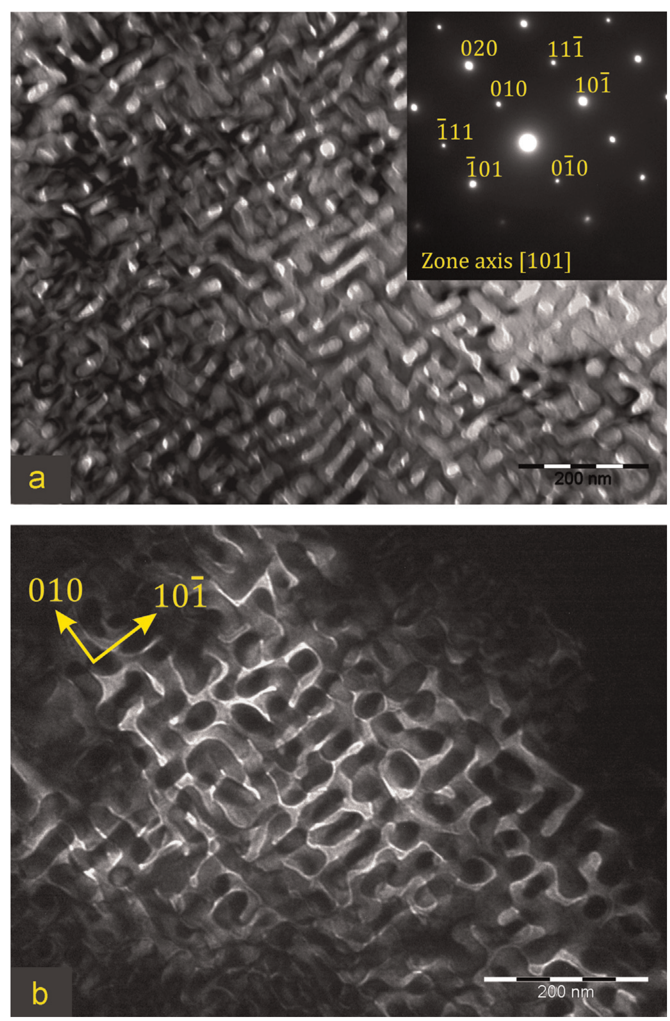

Fig. 1. Micrographs of Alni alloy cooled from 1240 °C at a critical rate: (a) bright-field image and (b) dark-field image taken in the 010 reflection of the B2 phase; (1a, inset), zone axis [101].