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Peculiarities of the spinodal decomposition and magnetic properties in melt-spun Fe_2NiAl alloy during aging

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ABSTRACT

A microcrystalline Alni alloy with the nominal composition $Fe_{51.1}Ni_{23.5}Al_{23.7}Si_{1.7}$ was prepared by melt-spinning. Detailed studies of the spinodal decomposition (SD), microstructure and magnetic properties of the ribbons subjected to isothermal aging at T_{ag}=500–780 °C are reported. TEM investigations of the decomposition of the solid solution ($\beta_2 \rightarrow \beta + \beta_2$) after melt-spinning and aging have revealed several types of decomposition products. As prepared melt spun ribbons show zone structure and antiphase domain (APD) boundaries inside grains. The APDs were formed during rapid solidification via nucleation and growth mechanism (A2–B2). Aging at 500–600 °C led to the coarsening of the zone microstructure and to the appearance of discontinuous precipitates (DP) ($\beta + \beta_2 \rightarrow \beta' + \beta'_2$) at grain boundaries. Aging at higher than 650 °C causes the formation of a modulated microstructure instead of a zone microstructure inside grains and coarsening of DP products. As-prepared ribbons did not reveal hard magnetic properties (H_c=6 Oe). The increase in H_c was observed with increasing aging temperature. The maximum coercivity H_c=250 Oe was obtained for the ribbons aged at 700 °C.

1. Introduction

Hard magnetic materials based on as-cast Fe–Ni–Al–Co alloys have attracted a considerable interest from both, the scientific and technical point of view [1] because of their high coercivity values arising due to spinodal decomposition (SD) of the solid solution ($\beta_2 \rightarrow \beta + \beta_2$). The coercive force of these type alloys depends on the magnetization difference between β and β_2 phases as well as the shape anisotropy in the ferromagnetic β phase. Recent studies of Alni and Alnico alloys [2–7] gave new results on SD products, ordering and chemical compositions of β and β_2 phases which show that the magnetic properties of these alloys could be further improved [3,4]. The properties of melt spun FeNiAl-based ribbons distinct from those of as-cast alloys. The microstructures, as well as the magnetic properties of the ribbons are largely determined by the cooling rate used to solidify from the melt and temperatures of subsequent aging [6,7]. The grain boundaries (GB) and antiphase domain (APD) boundaries are markedly affected by the SD products. A discontinuous precipitation (DP) was earlier revealed in Cu–Ni–Cr alloys [8–10] and the effect of GB on spinodal decomposition leading to the formation of DP at the GBs was studied using computer simulation [11–13]. A DP reaction had been observed for the first time in the Fe_2NiAl ribbons after subsequent aging in our work [7], in which the cellular microstructure at GBs consisting of alternating lamellas of β'- and β'_2 phases was sighted after aging the ribbons at 500 °C. However, the spinodal transformations and its influence on magnetic properties during aging at higher temperatures has not been carried out, although it is need for an understanding the formation of high coercivity state in AlNi-based alloys.

In objective of this study is to present new data related to the microstructure transformations and coercive force in the melt spun Alni alloy after isothermal aging at different temperatures. The peculiarities of the spinodal decomposition on the GBs and APD boundaries and the DP reaction development at the GBs during the aging at the temperatures 500–800 °C are considered and the relationship between microstructure and coercivity is briefly discussed.

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 cast ingot was melted and quenched by melt-spinning with a copper-wheel speed $V \sim 40$ m/s. The thickness of the melt spun ribbons was 20–40 μm. The aging of ribbons was carried out in an argon atmosphere at 500–780 °C for 10 min. The

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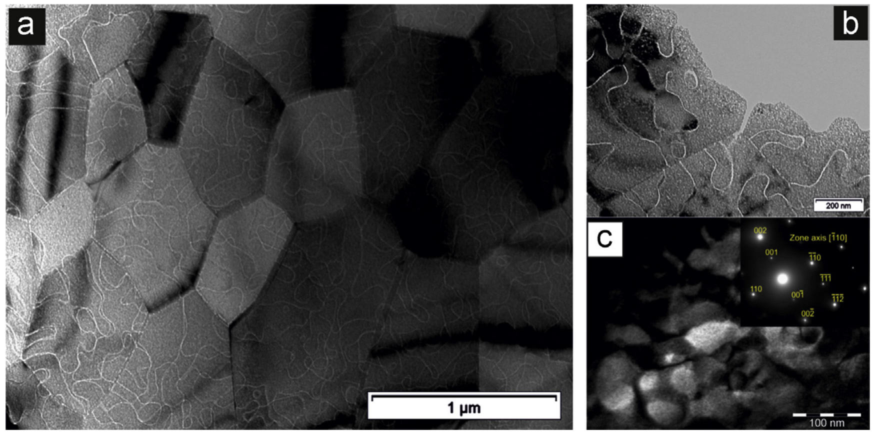

Fig. 1. Brightfield TEM micrographs of Alni ribbon, at low (a) and higher magnification (b), and (c) darkfield image taken in 001 reflection and subsequent diffraction pattern (inset).

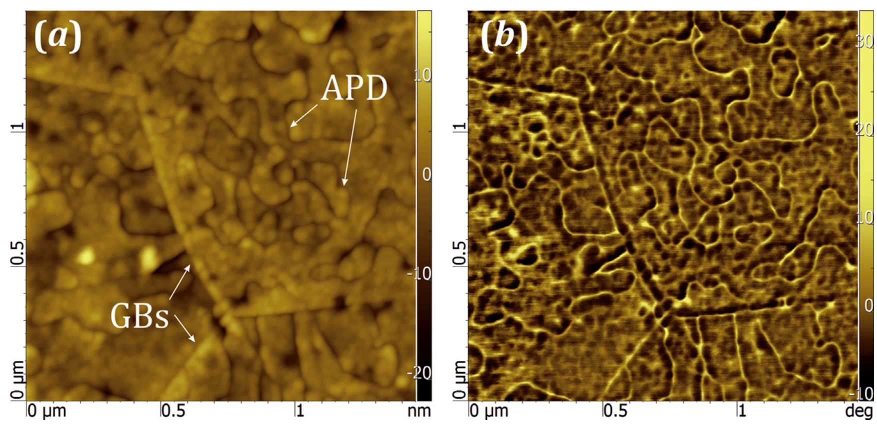

Fig. 2. AFM image of the ribbon microrelief in (a) height and (b) phase signals.

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 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 electropolishing. Note, that Fe-rich β phase dissolves rather than NiAl-rich β_2 phase during the electropolishing, that is confirmed by the chemical composition data which were measured at the late stages of decomposition. The microrelief was studied by an atomic force microscope of AIST-NT manufacturer in a taping mode. The magnetic properties of the ribbons were measured at room temperature using a PPMS EverCool-II (Quantum Design) magnetometer.

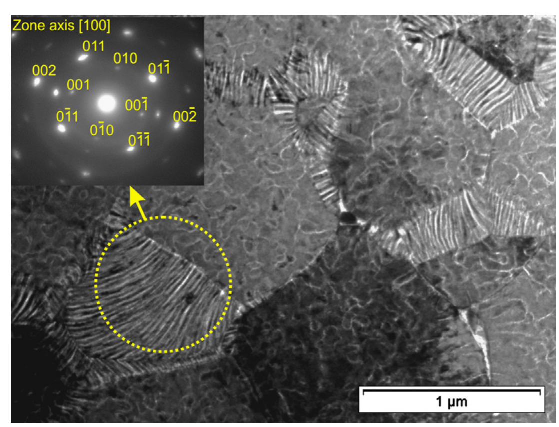

Fig. 3. A brightfield TEM micrograph of the ribbon aged at 550 °C and diffraction pattern correspond to the DP area.

3. Results and discussion

Fig. 1 shows (a and b) bright- and (c) dark-field images of the microstructure of Alni ribbon quenched from the melt and (1c, inset) associated electron diffraction pattern taken in 001 superlattice reflection. As-prepared ribbons do not exhibit hard magnetic properties (H_c=6 Oe).

In accordance with our previous work [7], the microstructure of Alni ribbon does not conform to the homogeneous solid-solution, and zone structure which corresponds to partial decomposition of solid solution into β and β_2 phases within the miscibility gap was observed. The twisted lines correspond to positions of APD boundaries, which are decorated by precipitates of decomposition products. The anti-phase domains are also observed in 001 reflection (Fig. 1c) because the electron phase shift is not zero (displacement vector for APD in B2 is a/2 < 111 >). The sequential ordering took place during the spinning of the alloy and APDs were formed during solidification from the melt via nucleation and growth mechanism (transformation of disordered