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Investigation of temperature dependent magnetization and elastic modulus in Si-doped $Fe_{65}Ni_{35}$ invar alloys

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ABSTRACT

Fe_{65} Ni_{35} invar is a prominent nickel steel alloy exhibiting low coefficient of thermal expansion around room temperature. The present work deals with the temperature dependence of the elastic modulus and magnetization of Si-doped invar alloys. X-ray diffraction measurements and scanning electron microscopy studies are performed for the structural characterization of the alloys. The elastic modulus of Fe_{65} Ni_{35} and Si-doped, Fe_{65} Ni_{35-x} Si_x samples of three different compositions with $0.55 \le x \le 2.5$, are measured in temperature range $23 \le T \le 600^{\circ}$C by the impulse excitation technique. The slope of the elastic modulus with respect to temperature for the Si-doped invar alloys are compared with the known values of non-doped invar. The temperature dependence of the magnetization and elastic modulus are compared.

1. Introduction

Fe_{65} Ni_{35} invar is an important steel alloy due to the low coefficient of thermal expansion (CTE) around room temperature [1]. It is used in applications where dimensional stability and precise positioning under changing temperature and load conditions are required as in motor valves, shadow-mask frames and seismic creep gauges [2]. Fe_{65} Ni_{35} has a face centered cubic (fcc) structure around room temperature. In addition to the well-known CTE anomaly, Fe-Ni invar alloys also exhibit anomalous features in the temperature dependencies of the magnetization, heat capacity, and atomic volume. Invar anomalies begin to be observed in a compositional range which is very close to the onset of the martensitic transition occurring from the fcc to the body centered cubic (bcc) structure. For a common material, when the temperature increases, the volume increases and the elastic modulus E decreases. However, the invar alloy exhibits anomalies around room temperature, whereby increasing the temperature leads to an increase in E. The positive volume-magnetostriction in the invar alloy compensates the volume shrinkage with decreasing temperature so that the thermal expansion remains almost constant. Also, the crystal softens so that the elastic constants increase with increasing temperature around Curie temperature T_c [3]. This anomalous volume-magnetostriction property of the invar alloy is a consequence of high-spin/low-spin transition caused by thermal moment-volume fluctuations [4,5]. Since the elastic modulus of a material is a measure of the interatomic binding strength, the invar anomalies with volume-dependent binding energies are also reflected in the elastic properties. To understand the origin of the invar anomalies, numerous works have been performed on the behavior of physical parameters as a function of composition, temperature and pressure [6–10].

Fe-Ni and Fe-Si alloys are of importance in the iron-steel industry due to the fact that they are widely used as soft magnetic materials with high electrical resistivity and high magnetic permeability [11]. Furthermore, the Fe-Ni-Si ternary system carries much importance because of the good oxidation resistance and high strength [12]. There are several studies on Si doped Fe-Ni alloys, however, none of them involve compositions around the invar composition and deal with invar anomalies in the elastic modulus and magnetization [13,14].

In this work, Si-doped Fe-Ni alloys are investigated in relation to their structure, elastic properties and magnetic properties. The results are compared with those of the well-known Fe_{65} Ni_{35} invar alloy.

2. Experimental

Fe_{65} Ni_{35} and Fe_{65} Ni_{35-x} Si_x alloys with $x = 0.55$, 1.28 and 2.5 at% were produced in an arc furnace using high purity elements (99.9%). For homogenization, the samples were annealed under Ar at 1073 K in sealed quartz tubes for 5 days. To verify the sample homogeneity, EDX

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Table 1
Compositions determined by EDX of the nominal Fe_{65} Ni_{35-x} Si_x series with less than 0.1% EDX absolute accuracy.

Fe (at%)	Ni (at%)	Si (x) (at%)
65	35	0
65	34.45	0.55
65	33.72	1.28
65	32.5	2.50

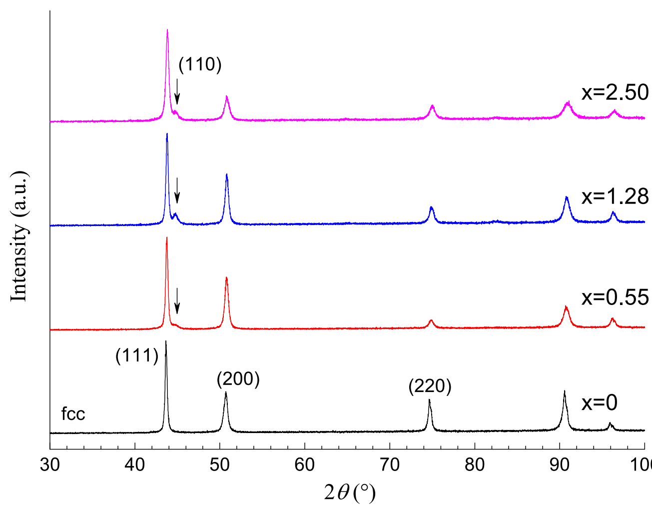

Fig. 1. X-ray diffraction patterns shown in the range $30^\circ \le 2\theta \le 100^\circ$. Black, red, blue and magenta data correspond to the samples with $x = 0$, 0.55, 1.28, and 2.50 at%, respectively. The arrows indicate the position of the (110) peak of Fe_2 Si in the Si-doped samples. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

spectra were collected from three different areas. The compositions were found to be homogeneous to within 0.05% for all samples. The compositions of the 4 samples are listed in Table 1. Scanning electron microscopy (SEM) images were obtained on polished and subsequently etched specimens. X-ray diffraction (XRD) measurements were carried out in the 2θ range $20^\circ \le 2\theta \le 120^\circ$ using a Cu K_α x-ray source. Magnetization measurements were made using a super conducting quantum interference device magnetometer equipped with an oven attachment. The impulse excitation technique (IET) was used for the elastic modulus measurements [15]. Different from measuring standard stress-strain curves, IET uses the internal friction corresponding to an impulse excited resonant frequency to determine E. The IET measurements are gaining importance in determining E, shear modulus, and damping parameters as a consequence of the method being non-destructive and relatively faster than other conventional methods.

3. Results

In the following, we present the results on x-ray diffraction (XRD), scanning electron microscopy (SEM), temperature-dependent magnetization $M(T)$, and temperature-dependent elastic modulus $E(T)$ measurements. Firstly, the results of XRD and SEM studies are introduced to understand the crystallographic structures of the samples. Room temperature XRD measurements are given in Fig. 1 in the range $30^\circ \le 2\theta \le 100^\circ$. As seen from the black diffractogram, the invar alloy without Si exhibits characteristic peaks of the fcc structure. However, a small amount of Si-doping leads to the appearance of Fe_2 Si in addition to the cubic Fe_{65} Ni_{35} phase. The peak related to this structure at 44.85° labelled as (110) is observed in all Si-doped samples.

SEM images of all samples are given in Fig. 2a–d. For the sample $x = 0$ at% in Fig. 2a, grains of the fcc austenite structure are observed as expected. When small amount of Si are added, the

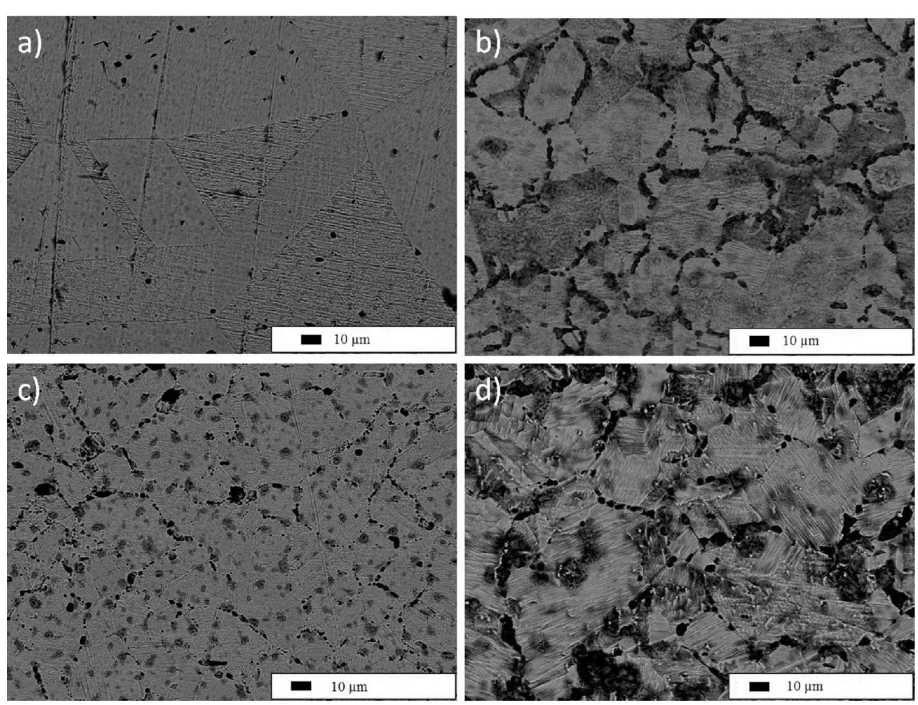

Fig. 2. Scanning electron microscopy images of the samples (a) $x = 0$, (b) $x = 0.55$, (c) $x = 1.28$, and (d) $x = 2.50$ at%.