



Magnetic and transport studies of σ -phase Fe₅₀V₅₀ alloys with different thermal history

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ABSTRACT

The influence of the formation conditions of the sigma phase in an equiatomic FeV alloy on the magnetic and electric transport properties is studied. It is found that a sigma phase sample with higher resistivity (subject to a previous long annealing) has a much sharper magnetic transition than one formed after a shorter heat treatment and quenching, although both have very similar magnetic moments and ferromagnetic transition temperatures ($T_c \sim 15$ K from minimum dM/dT).

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1. Introduction and experimental

The hard and brittle sigma phase forms at elevated temperature in various alloy systems involving transition elements. The sigma phase forms in FeV alloys in wide temperature and composition ranges. At high temperature, the equiatomic alpha-FeV alloy may transform into a metastable ordered phase having a B2 type structure prior to the formation of the sigma phase. In previous studies the decrease of the electrical resistivity in a Fe₅₀V₅₀ alloy with holding temperatures at 1015 and 1115 K was suggested to result from a possible order of the alpha phase, namely the formation of a transient B2 structure, in agreement with literature results [1–3]. The local Fe/V order influences the magnetic properties of the alloy. As an example, Krause et al. [4] have found that the magnetic moment is almost zero in slowly cooled Fe–V alloys with V content above 45% while a ferromagnetic phase is observed in samples quenched at low temperature.

A Fe₅₀V₅₀ alloy was prepared by melting together the elements in an arc-melting furnace. As obtained, the alloy consists of alpha phase. The samples of study of the present work, named S3 and S4 were cut from the ingot to perform subsequent heat treatments.

Sample S4 sample was annealed at 1300 K for 4 days to be transformed into the sigma phase. The samples (S3 in alpha phase and S4 in sigma phase) were then heated in argon from RT to 1000 K and then cooled down to RT immediately, by letting the furnace cool. The heating rates were 10 K/min. Electrical resistivity (ρ) measurements were performed in situ during thermal treatments of specimens in flowing argon, by a conventional ac four probe method. To characterize the samples before heating and after cooling, X-ray diffraction (XRD) studies were performed at RT using CuK α radiation ($\lambda = 0.17889$ nm) and RT ⁵⁷Fe Mössbauer (MS) spectra were recorded in a conventional constant acceleration transmission geometry. Magnetization (M) was measured in a vibrating sample magnetometer (Cryogenic Ltd.) in the temperature range 4–300 K; maximum field was $H = 100$ kOe.

2. Results and discussion

We studied previously the transformations of Fe₅₀V₅₀ from alpha to sigma phase with different heat treatments using several techniques. Almost pure sigma phase sample was obtained with the described heating up to 1000 K of the sample S3 followed by immediate cooling. In this work, the sigma phase sample (S4) was subjected to the same heating–cooling cycle. The results of the in situ electrical resistivity measurements of samples S3 and

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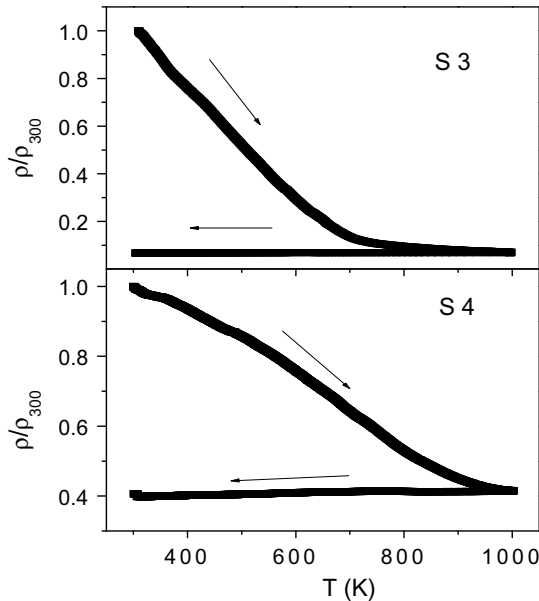


Fig. 1. Electrical resistivity relative to the resistivity at RT, as a function of temperature for S3 and S4 samples heated up to 1000 K and cooled immediately.

S4 during the thermal cycles are presented in Fig. 1. The initial ($T = 300$ K) electrical resistance of sample S3 is 0.5Ω (alpha phase), while for sample S4 it is 70Ω (sigma phase). In both cases, one observes a considerable decrease of resistivity with temperature in heating. The rapid cooling quenches the structure obtained, with resistance changes of 2% and 4% on cooling, respectively, for S3 and S4. The large difference in resistance is mostly due to the difference in phase, since their dimensions and contact distances were very similar. Unfortunately, it was not possible to measure the absolute resistivity of the samples.

MS and XRD confirm that in S3 the transformation from alpha to sigma phase occurred and that in sample S4 the sigma phase was kept. The mean hyperfine field of the starting and remaining alpha phase after the heat treatments of the samples is about 26 T. The mean isomer shift of samples S3 and S4 after treatments is $-0.21(1)$ and $-0.23(1)$ mm/s, respectively. These values are close to the characteristic value of the sigma phase for nearly-equiatomic FeV alloys, so no great information can be obtained on the atomic ordering of the two samples. It has been shown that for sigma FeCr alloys the isomer shift depends on the stage of the transformation from α to σ [5]. In the present case no great difference is observed in the isomer shift that merits some conclusions.

From MS shown in Fig. 2 one obtains a residual alpha phase of 6% in S3 and 4% in S4. The magnetization (M) isothermals at 50 K for S3 and S4 (Fig. 3) show the same behavior up to 100 kOe and confirm also that the amount of alpha phase is residual [1].

The temperature dependence of the magnetization (at $H = 198$ Oe) of samples S3 and S4 is presented in Fig. 4. The strong increase of magnetization on cooling results from the magnetic ordering at low temperatures. The high temperature behavior (almost plateau) is due to the residual alpha phase (ferromagnetic above RT). While in sample S4 the transition is relatively sharp ($T_c \sim 15$ K), in sample S3 one observes a much broader phase transition, spreading from 60 to 10 K. The inset presents the temperature derivative dM/dT : although in both samples the minimum of dM/dT occurs at close temperatures, the deep for S3 is much broader.

Our results indicate that although the applied heat treatments resulted in a sigma phase structure, there might be some differences in the local atomic Fe and V site occupancies in S3 and S4.

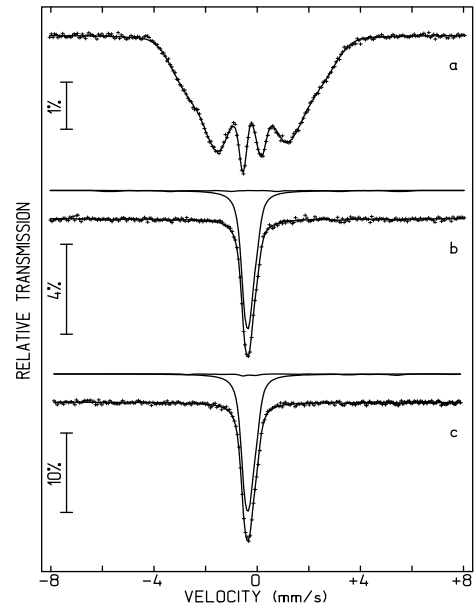


Fig. 2. Mössbauer spectra of the samples (a) before heat treatments (alpha phase), after temperature cycling; (b) S3 sample and (c) S4 sample.

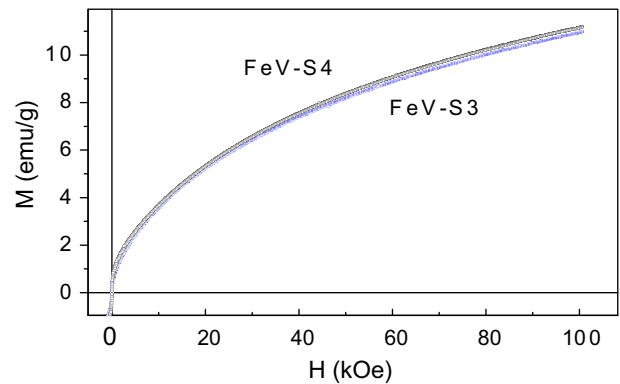


Fig. 3. Magnetization isotherms at $T = 50$ K.

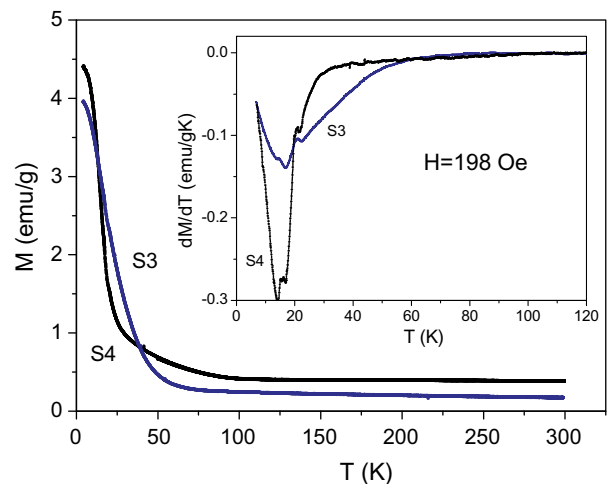


Fig. 4. Temperature dependence of the magnetization M for S3 and S4 (inset: temperature derivatives dM/dT).

In fact, the magnetic field dependence in the paramagnetic phase (at 50 K, shown in Fig. 3) reveals no difference, as only the average

global scale behavior is relevant. On the other hand, the local order influences critically the magnetic interactions (low temperature dependence) and the electrical resistivity.

One expects that S4 has much more random site occupancies, since it was annealed for long time before this heat cycling. This resulted in a much higher resistance of S4 compared to S3. This randomness is homogeneous, resulting in a sharper magnetic transition, related to the average magnetic interactions. Sample S3, in which the sigma phase was formed during this heat cycling, the different short range order of Fe–Fe/Fe–V neighbors is responsible for the large distribution of magnetic interactions. We associate this difference to the influence of the transient metastable ordered phase having a B2 type structure (particularly strong for an equiatomic FeV alloy), prior to the formation of the sigma phase.

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