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Kinetics of the σ -phase formation in an $(\text{Fe}_{53.8}\text{Cr}_{46.2})\text{--}0.1 \text{ at.}\% \text{ Ti}$ alloy

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ABSTRACT

The kinetics of the σ -phase formation promoted by an isothermal anneal at $T = 700^\circ\text{C}$ has been studied in a polycrystalline alloy of $(\text{Fe}_{53.8}\text{Cr}_{46.2})\text{--}0.1 \text{ at.}\% \text{ Ti}$ using the ^{57}Fe Mössbauer effect. It was found that the addition of $0.1 \text{ at.}\% \text{ Ti}$ to an $\text{Fe}_{53.8}\text{Cr}_{46.2}$ alloy drastically accelerates the $\alpha\text{--}\sigma$ transformation. The fraction of the σ phase precipitated follows the Avrami–Johnson–Mehl equation. The best fits to the data yielded the time constants, from which a Ti-induced decrease in the activation energy by about 14.0 kJ mol^{-1} was estimated.

The σ -phase, which occurs in many transition-metal alloys, is of a great interest both for technological and for scientific reasons. The former stems from the fact that its presence causes materials properties to deteriorate significantly, while the latter is related to gathering knowledge of the physical properties of the phase. One of the alloy systems in which the presence of the σ -phase is of a particular importance is Fe–Cr. This follows, firstly, from the role the alloy plays in the steel making industry and, secondly, from its unusual magnetic properties. Consequently, Fe–Cr alloy has been regarded as a model system in various studies. It can be 100% transformed into the σ phase by annealing provided that the Cr content lies within the range 45–50 at.% and the annealing temperature T in the range $773 \text{ K} \leq T \leq 1110 \text{ K}$ (Kubaschewski 1982). Addition of the so-called steel-making elements, for example Mn, Mo, Si, Co, Nb, V or W, is believed not only to accelerate the formation of the phase but, in addition, to influence the range of its existence in the phase diagram (Hall and Algie 1966). However, detailed studies of the issue are yet to be done. On the other hand, addition of tin into an Fe–Cr alloy was found to retard the formation of the phase (Costa and Dubiel 1993).

In this letter we report the kinetics of the $\alpha\text{--}\sigma$ phase transformation in a polycrystalline alloy of $(\text{Fe}_{53.8}\text{Cr}_{46.2})\text{--}0.1 \text{ at.}\% \text{ Ti}$. The alloy was prepared by melting appropriate amounts of Fe (99.9% purity), Cr (99.9% purity) and Ti (99.9% purity) in a vacuum induction furnace. The received ingots were cut into cubes of

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Figure 1. Room-temperature ^{57}Fe Mössbauer spectra recorded on an $(\text{Fe}_{53.8}\text{Cr}_{46.2})$ -0.1 at.% Ti sample (a) as quenched (100% α) (b) annealed at $T = 700^\circ\text{C}$ for $t = 6\text{ h}$ (17% σ), (c) annealed at $T = 700^\circ\text{C}$ for $t = 16\text{ h}$ (57% σ) and (d) annealed at $T = 700^\circ\text{C}$ for $t = 48\text{ h}$ (100% σ): (—), best fits to the data; (·····), subspectrum of the σ phase.

5 mm $\times$ 5 mm $\times$ 5 mm size, which were subsequently homogenized by vacuum annealing for 24 h at $T = 1200^\circ\text{C}$ followed by water quenching. The chemical composition of the homogenized sample was determined by electron probe micro-analysis. The concentrations of Fe, Cr and Ti were evaluated as an average over five values found in various places across the sample (the values for Ti were within the range 0.04–0.18). The transformation from the α into the σ phase was carried out by an isothermal anneal in vacuum at $T = 700^\circ\text{C}$. The kinetics of the transformation were studied by means of ^{57}Fe Mössbauer spectroscopy. Room-temperature spectra were recorded in a transmission geometry using a standard spectrometer and a ^{57}Co -Rh source of about 20 mCi activity. The samples for the Mössbauer measurements were in the form of powder obtained by filing the cubes with a diamond file.

Examples of the spectra can be seen in figure 1. The distinction between the α and the σ phases can be based on their Curie temperatures T_C . T_C for the α phase is much higher than 295 K; hence the spectrum recorded at 295 K for this phase shows features characteristic of a magnetic sample (six-line pattern). T_C for the σ phase is much lower than 295 K, and consequently its recorded spectrum has the form characteristic of a non-magnetic sample. The fraction A_σ of the σ phase was determined from the spectral area of the subspectrum ascribed to the σ phase, assuming the Lamb-Mössbauer factors had the same values for the two phases. The spectral area was determined by fitting the spectra with a least-squares procedure assuming a distribution of spectral parameters, of the hyperfine field for the magnetic subspectrum, and of the isomer shift for the non-magnetic subspectrum. The A_σ values obtained in this way are displayed in figure 2 against annealing time t , both for Ti-doped (full circles) and undoped (open circles) samples. It is evident that the presence of Ti in the studied sample drastically accelerates the formation of the σ phase. While annealing for about 170 h was required to transform the undoped sample 100% into the σ phase, annealing for only about 30 h was required to do the

Figure 2. The fraction A_σ of the σ -phase in the $\text{Fe}_{53.8}\text{Cr}_{46.2}$ (○) and $(\text{Fe}_{53.8}\text{Cr}_{46.2})\text{-}0.1\text{ at.\% Ti}$ alloys (●) as a function of annealing time t .

same for the doped sample. The solid curves represent the best fits to the data in terms of the Avrami–Johnson–Mehl equation

$$A_\sigma = 1 - \exp[-(kt)^n], \tag{1}$$

where k is a time constant and n is the form factor. The best-fit values are presented in table 1. They give clear evidence that in both samples the growth of the σ phase is controlled by a diffusion process, in which particles of this phase nucleate at a constant rate (Guy 1972). The time constant for the Ti-doped sample is about 6 times larger. From the results obtained, one can also estimate a difference in the activation energy for the two samples studied. For this purpose, the following equation was used:

$$k = k_0 \exp\left(-\frac{E}{k_B T}\right), \tag{2}$$

where E is the activation energy and k_B Boltzmann’s constant. Using for k the values from table 1 and assuming for both samples the same value for k_0 , one arrives at $\Delta E = 14.0 \text{ kJ mol}^{-1}$ (ΔE being the difference between E for the undoped and E for the doped sample). Taking further into account the activation energy for ^{51}Cr in $\alpha\text{-Fe}$ to be $240.7 \text{ kJ mol}^{-1}$ (Weast 1985), one arrives at $E = 226.7 \text{ kJ mol}^{-1}$ for the $(\text{Fe}_{53.8}\text{Cr}_{46.2})\text{-}0.1 \text{ at.\% Ti}$ alloy. It is worth noting that $E = 106.2 \text{ kJ mol}^{-1}$ was found for an $\text{Fe}_{53.7}\text{Cr}_{42.1}\text{Mo}_{4.2}$ alloy (Waanders *et al.* 1998).

Table 1. The best-fit values for k and n parameters in the Avrami–Johnson–Mehl equation.

Sample	k (h^{-1})	n
$\text{Fe}_{53.8}\text{Cr}_{46.2}$	11.7×10^{-3}	2.6
$(\text{Fe}_{53.8}\text{Cr}_{46.2})\text{-}0.1 \text{ at.\% Ti}$	64.5×10^{-3}	2.4

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