



	Experiment title: Kinetics of the σ -phase decomposition	Experiment number: MA-398
Beamline: ID18	Date of experiment: from: 21.01.2008 to: 25.01.2008	Date of report:
Shifts: 12	Local contact(s): Dr. Rudolf RUEFFER	<i>Received at ESRF:</i>
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Report:

The σ -phase in the Fe-Cr system forms in a narrow range of concentrations, viz. $50 < x < 55$ at%. It precipitates from the α -phase (bcc) during an isothermal annealing in the temperature range of $\sim 530 < T < \sim 830^\circ\text{C}$. The transformation rate depends on temperature being maximal at 700°C . Below $\sim 530^\circ\text{C}$ the σ -phase is stable. Above $\sim 830^\circ\text{C}$ it dissolves to the α -phase. The process of the α - σ transformation was intensively investigated, whereas there is nearly no information available on the reversed i.e. σ - α transformation, which according to the phase diagram should take place at $T \geq 830^\circ\text{C}$. The mechanism and the kinetics of the transformation are the subject of this work.

The kinetics should be measured both versus temperature and alloy composition. Three highly ^{57}Fe enriched samples with compositions $\text{Fe}_{55}\text{Cr}_{45}$, $\text{Fe}_{53}\text{Cr}_{47}$ and $\text{Fe}_{51}\text{Cr}_{49}$ were prepared. The nuclear resonant scattering spectra measured in forward direction at the beamline ID18 turned out to be different enough to distinguish between the σ and α phases and to analyse their relative amounts. Unfortunately not enough spectra could be measured due to technical problems and time limitations but our measurements have been completed by the traditional Mossbauer spectroscopy.

The kinetics were measured in constant temperatures chosen for each sample separately. The example family of kinetics for $\text{Fe}_{51}\text{Cr}_{49}$ is presented in Fig. 1. The σ - α transformation process starts below 825°C for this sample. It is extremely slow in this temperature (after 7 hours the transformation is not finished yet), whereas for 830°C it took only 1.5 hour. The transformation rate increases quickly with increasing temperature and for the highest measured temperature the time of transformation is shorter than 4 minutes. All the kinetics were successfully described in terms of the Johnson-Avrami-Mehl approach:

$$A = A_0(1 - \exp(-kt^n)),$$

where k is a time constant and n is the form factor. It gives clear evidence that the σ - α transformation is

controlled by a diffusion process, like in case of the α - σ conversion. Based on the results obtained, one can also estimate the activation energy for this sample (Fig. 2). For this purpose, the following equation was used:

$$k = k_0 \exp(-E/k_B T),$$

where E is the activation energy and k_B the Boltzman's constant. Using for k the values obtained in the above-mentioned analysis, one arrives at $E_{\sigma-\alpha} = 13.1\text{eV}$. This value is considerably higher than the corresponding value of $E_{\alpha-\sigma} = 2.1\text{eV}$ for the α - σ transformation. Such a great difference between the activation energies of the α - σ and σ - α transformations seems to be in line with the fact, that the former conversion occurs in much higher temperature than the later one.

The measurements of the σ - α transformation kinetics for remaining two samples are in progress. They will allow us to determine the activation energy values, as well as to verify different kinetics models.

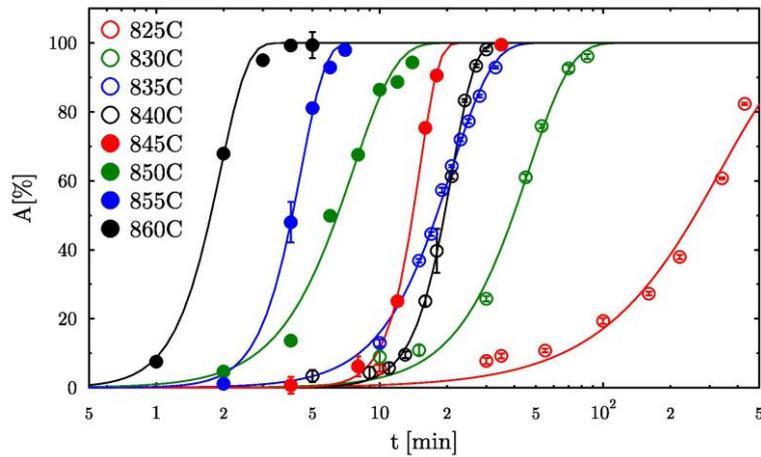


Fig. 1. Kinetics of the σ - α conversion for the $\text{Fe}_{51}\text{Cr}_{49}$ sample. Solid lines represent best fits according to Johnson-Avrami-Mehl equation. Transformation temperatures are indicated.

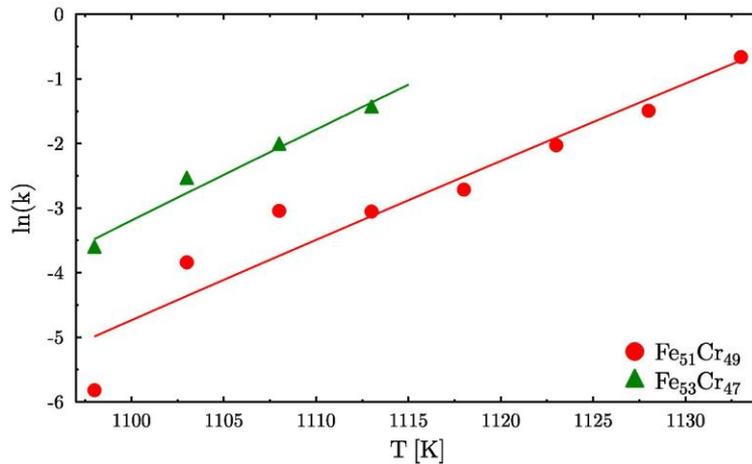


Fig. 2. Time constants of the σ - α conversion for the $\text{Fe}_{51}\text{Cr}_{49}$ and $\text{Fe}_{53}\text{Cr}_{47}$ samples. Solid lines represent best fits to the data.