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# Experiment Report Form

**The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF**.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

*http://193.49.43.2:8080/smis/servlet/UserUtils?start*

***Reports supporting requests for additional beam time***

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

***Reports on experiments relating to long term projects***

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

***Published papers***

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

## Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

## Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

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


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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 ~530 < T < ~830$^{O}$C. The transformation rate depends on temperature being maximal at 700$^{O}$C. Below ~530$^{O}$C the σ-phase is stable. Above ~830$^{O}$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≥830$^{O}$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 $Fe_{55}Cr_{45}$, $Fe_{53}Cr_{47}$ and $Fe_{51}Cr_{49}$ 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 Mössbauer spectroscopy.

The kinetics were measured in constant temperatures chosen for each sample separately. The example family of kinetics for $Fe_{51}Cr_{49}$ is presented in Fig. 1. The σ-α transformation process starts below 825 $^{O}$C for this sample. It is extremely slow in this temperature (after 7 hours the transformation is not finished yet), whereas for 830$^{O}$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 then 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:

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

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 $E_{\sigma\text{-}\alpha}$ = 13.1eV. This value is considerably higher than the corresponding value of $E_{\alpha\text{-}\sigma}$ = 2.1eV 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.

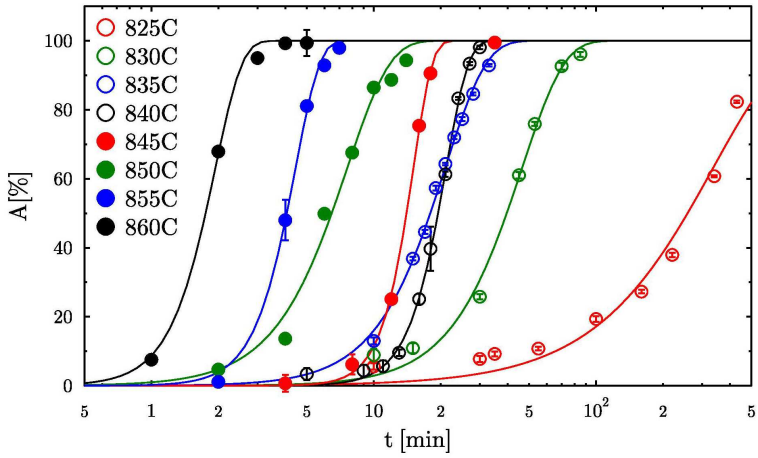


Fig. 1. *Kinetics of the σ-α conversion for the $Fe_{51}Cr_{49}$ sample. Solid lines represent best fits according to Johnson-Avrami-Mehl equation. Transformation temperatures are indicated.*

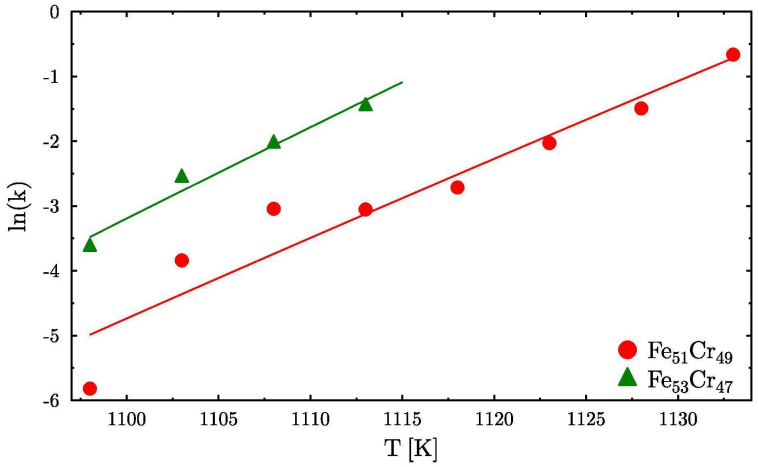


Fig. 2. *Time constants of the σ-α conversion for the $Fe_{51}Cr_{49}$ and $Fe_{53}Cr_{47}$ samples. Solid lines represent best fits to the data.*