ESRF	**Experiment title:** In-situ study of the nucleation and growth of individual grains during the austenite-ferrite phase transformation in steel.	**Experiment number**: ME-176
Beamline: ID11	**Date of experiment**: from: 18-june-2001 to: 23-june-2001	**Date of report**: 27-Feb-2002
Shifts: 11	**Local contact(s)**: Dr. S. Grigull	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

S.E. Offerman[1,2,*], N.H. van Dijk[2,*], M.Th. Rekveldt[2], J. Sietsma[1,*], S. van der Zwaag[1], L. Margulies[3,4,*], and S. Grigull[4,*]

1: Laboratory of Materials Science, Delft University of Technology, 2628 AL Delft, The Netherlands
2: Interfaculty Reactor Institute, Delft University of Technology, 2629 JB Delft, The Netherlands
3: Materials Research Department, Risø National Laboratory, DK-4000 Roskilde, Denmark
4: European Synchrotron Radiation Facility, BP 220, F-38043 Grenoble Cedex, France

Report:

A long-standing problem in the field of materials science is the modeling of the austenite-ferrite transformation kinetics in steel. This phase transformation plays a crucial role in the formation of microstructure, which in turn governs the mechanical properties. The first attempts to model the austenite-ferrite transformation date back several decades and are described by the classical Johnson-Mehl-Avrami theory [1,2]. Despite continuous efforts in recent years [3] the predictive power of these models is still limited as they strongly depend on the physical properties on a microscopic scale, which are not sufficiently known.

In order to get a better insight in the transformation kinetics in the bulk of the material in-situ experimental techniques at high temperatures (1000-1200 K) are required. So far, the fraction transformed has been studied by thermal expansion measurements [3], while 3D neutron-depolarisation measurements [4,5] give additional information on the *average* ferrite grain size below the ferromagnetic Curie temperature of ferrite (T_C = 1043 K). X-ray diffraction measurements at high temperatures, using the 3DXRD microscope [6], takes the characterization one step further and provides simultaneous measurements of the nucleation and growth of several *individual* ferrite grains *in-situ*.

The aim of the experiments was to identify the physical parameters that characterize the nucleation and growth mechanism of ferrite grains in the austenite matrix during isothermal transformation and continuous cooling experiments. The transformation behaviour of three medium carbon steels was studied, with carbon concentrations of 0.22, 0.35 and 0.45 wt%. From the growth behavior of the individual grains valuable information was deduced regarding the incubation time for nucleation, nucleation density, growth rate, interface mobilities, and preferred orientation as a function of time, transformation temperature, and carbon concentration.

In order to study the grain volume of individual ferrite grains as a function of time we used the diffraction instrument ID11 in transmission geometry with a 2D detector and a photon energy of 80 keV. For a low ferrite grain density the Bragg peaks of the individual ferrite grains appeared as separate spots on the 2D detector. The diffracted intensity is proportional to the grain volume. The position on the detector gives information about the orientation of the grain. To prevent that the spots overlap, a limited number of grains was illuminated by choosing an appropriate beamsize of 63×70 μm^2.

For the analysis of the data we have a collaboration with E.M. Lauridsen and H.F. Poulsen of the Risø National Laboratory in Denmark. Here we present the first results of measurements on a 0.35 wt% carbon steel, which was heated to 900 ^oC and held there for approximately 12 min. in order to form the austenite phase. The steel was subsequently rapidly cooled at 20 ^oC/s to the two phase region, where the transformation to ferrite started. The measured ferrite grain volume was converted to the grain radius by assuming that the ferrite grain is spherical. Figure 1 shows the grain radius of 7 individual grains as a function of time. More grains were analysed but are not shown in the figure. From this figure it can be seen that the incubation time is different for the individual grains and can be as large as appr. 8 min. Figure 2 shows the growth behaviour of the same grains. It is striking that six out of the seven grains show a rapid increase in radius during the first moment of their existence. This rapid increase at the beginning is not in accordance with the frequently used Zener-model [7], which assumes a diffusional transformation behaviour. However, the diffusional growth is observed at longer times (see Fig.2).

In the near feature the other measurements will be analysed, which include isothermal transformation experiments at different temperatures and continuous cooling experiments for the three steel grades. The observed transformation behaviour will be compared with well-known phase transformation models, including our own transformation model [3].

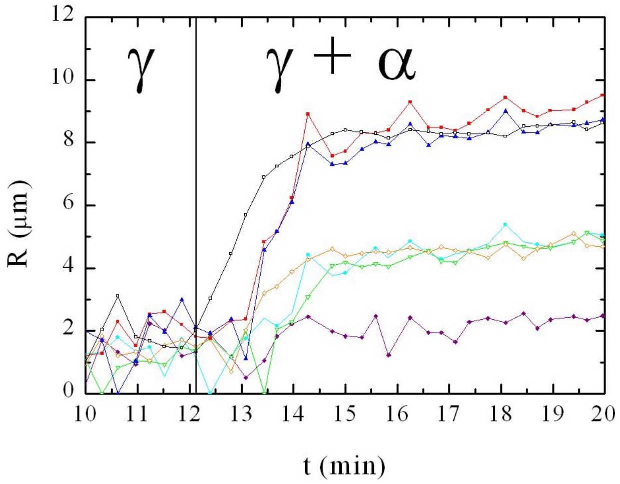

Fig. 1. *Nucleation and initial growth kinetics of individual ferrite grains during the isothermal γ/α-transformation of a C35 steel. The vertical line indicates the time at which the rapid cooling from the austenite stage to the two phase region started.*

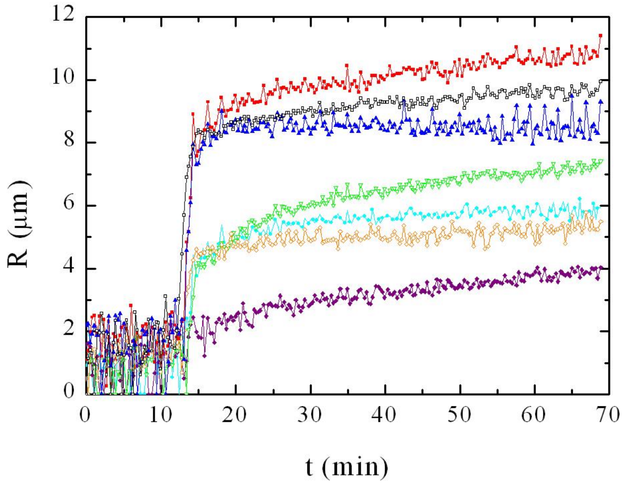

Fig. 2. *Entire growth curves of the 7 grains shown in fig. 1. From the growth curves it is clear that the main part of the phase-transformation takes place in the very early stages of the isothermal annealing. Notice the difference in size and growth behavior of the individual grains.*

[1] J. Johnson and R. Mehl, *Trans. A.I.M.E.* **135** (1939) 416.
[2] Avrami, *J. Chem. Phys.* **7** (1939) 1103; **8** (1940) 212; **9** (1941) 117.
[3] Y. van Leeuwen *et al.*, *Metall. Mater. Trans. A* **29A** (1998) 2925.
[4] S.G.E. te Velthuis *et al.*, *Physica B* **234-236** (1997) 1027
[5] S.G.E. te Velthuis *et al.*, *Acta Mater.* **48** (2000) 1105.
[6] E. M. Lauridsen *et al.*, *Scripta Mater.* **43** (2000) 561.
[7] C. Zener, *J. Appl Physics* **20** (1949) 950.