HIGH TEMPERATURE TEXTURE GONIOMETER FOR *IN SITU* MEASUREMENTS OF TRANSFORMATION TEXTURES

G. BRÜCKNER, F.R. REHER* and G. GOTTSTEIN

*Institut für Metallkunde und Metallphysik, RWTH Aachen,
Kopernikus Str. 14, D-52074 Aachen, Germany*

(Received 6 September 1996)

A high temperature stage was designed for mounting onto a computer controlled four circle X-ray texture goniometer. This technique allowed to conduct *in situ* texture measurement, i.e. the determination of the texture evolution during rather than subsequent to annealing. The device was employed for temperatures up to 1000°C.

The furnace consisted of a resistance wire of Pt30Rh, which was isolated against the specimen with Al₂O₃ glue. The furnace with the specimen was covered by a hemispherical KaptonTM foil. Inside the hemisphere a reducing gas atmosphere was used to avoid oxidation of the sample surface.

Keywords: Transformation texture; High temperature texture measurement

INTRODUCTION

The $\alpha \rightarrow \gamma$ phase transformation has been the subject of investigations for quite a long time, e.g. Bain published 'The Nature of Martensite' already in 1924 (Bain, 1924). The phase transformation of the most commercial steels occurs at high temperatures. Because of the experimental difficulties associated with the measurement of X-ray textures at elevated temperatures, there is virtually no information on austenite textures of commercial steel. For recording X-ray textures at high temperatures an existing four circle texture goniometer as described by

* Corresponding author

Hirsch *et al.* (1986) was modified (Reher *et al.*, 1994). This allowed the determination of textures at different temperatures in one sample and therefore detailed investigations of e.g. transformation textures.

CONSTRUCTION

The furnace was heated with a $\varnothing 0.5$ mm Pt30%Rh resistance wire, which allowed a wire temperature of up to 1500°C . The wire, bent to a spiral, was inserted in a ceramic body (Fig. 1). The density of the loops decreased towards the centre of the furnace in order to obtain a homogeneous specimen temperature. The electrical contact of sample and resistance element was prevented by covering the wire with Al_2O_3 glue in the ceramic body.

To avoid damage to the Euler cradle due to thermal expansion or degradation of the lubricating grease, the temperature of the cradle should not exceed 180°C . Therefore, the heat conduction to the Euler cradle was minimized by fastening the furnace onto low density ceramic pins, which were placed on aluminium cooling fins. This assembly kept the cradle temperature below 120°C even at maximum furnace power.

FIGURE 1 Schematic arrangement of the resistance element.

The specimen ($35 \times 24 \text{ mm}^2$) was clamped down onto the furnace with metal claws (Fig. 1). The measuring surface of the sample was $14 \times 24 \text{ mm}^2$. A Pt-Pt10Rh thermocouple, which measured the temperature in the heating spiral was connected to a temperature controller. This controller itself was programmed and monitored by a computer, which set the entire thermal cycle for the experiment. This design allowed a quick and easy specimen change without changing the thermocouple. Further optional thermocouples attached to the specimen were used to calibrate the furnace. Specimen temperatures in the centre and at the edge were measured and plotted as a function of the wire temperature in Fig. 2. The temperature gradients in the specimen were found to be less than 15°C . The temperature in the wire and in the specimen differed by about 250°C at maximum furnace power.

The furnace with the specimen was covered by a hemispherical KaptonTM foil with a thickness of $70 \mu\text{m}$. The hemisphere had been produced by deep drawing at 400°C . The spherical shape ensured a constant absorption of the X-ray beam for all angular positions. The intensity loss due to the foil was measured for different temperatures. In Fig. 3 the reflected intensities of the $\{110\}$ crystallographic planes were measured in a ϑ -scan with and without the foil at room temperature

FIGURE 2 Specimen temperature as a function of the heating wire temperature. Temperature gradients in the specimen did not exceed 15°C .

FIGURE 3 {110} reflection peak of low carbon steel with and without KaptonTM foil at $T=25^{\circ}\text{C}$, CoK_{α} -radiation. The intensity loss is about 10%.

with CoK_{α} -radiation. The X-ray intensity loss was found to be approximately 10%. The same result was obtained at 500°C .

Within the hemisphere a reducing gas atmosphere with a flux of about $5 \cdot 10^{-3} \text{ m}^3/\text{h}$ had to be used to avoid oxidation of the sample surface. For the first experiments a N_2 atmosphere was used. Because of the residual oxygen in the hemisphere the sample suffered strong oxidation, which could be measured in a ϑ -scan of the austenitic phase of a low carbon steel at 950°C (Fig. 4(a)). An addition of 5% H_2 reliably prevented an oxidation of the specimen (Fig. 4(b)).

In the Euler cradle the electrical wires and the gas supply of the hot stage were held in a U-shaped peripheral ring during the azimuthal rotation (Fig. 5). Every 360° the sense of rotation was reversed. For orthorhombic or higher sample symmetries the azimuthal rotation could be shortened to 90° , since only a quarter of the pole figure needed to be measured. This reduced the measurement time to 5 min for one pole figure and about 25 min for the determination of a full ODF (4 pole figures), respectively. This procedure could be useful for materials which undergo microstructural changes at high temperatures.

The reflected intensities could be recorded both by a scintillation detector and by a position sensitive detector. The latter is preferred when peak overlapping occur, e.g. in the ferrite + austenite region during the heating of carbon steels.

FIGURE 4 θ -scans for different gas atmospheres, $T=950^{\circ}\text{C}$, MoK_{α} -radiation. (a) N_2 -gas atmosphere; (b) $\text{N}_2 + 5\% \text{H}_2$ -gas atmosphere.

FIGURE 5 Hot furnace in the Euler cradle: the peripheral ring leads the tube and the wires during azimuthal rotation, the sense of rotation changes every 360° (See Color Plate 1).

SPECIFICATIONS

specimen temperature	: up to 1000°C
temperature gradients in the specimen	: $< 15^\circ\text{C}$
heating rate	: $150^\circ\text{C}/\text{min}$
specimen size	: $35 * 24 \text{ mm}^2$
measuring surface	: $14 * 24 \text{ mm}^2$
gas atmosphere	: N_2 with 5% H_2
X-ray intensity loss due to Kapton TM foil	: $\approx 10\%$
α -latitude	: $0^\circ - 85^\circ$
β -azimuth	: $0^\circ - 360^\circ$
angular velocity $\dot{\beta}$	: $6^\circ/\text{s}$
Bragg angle ϑ	: $5^\circ - 56^\circ$

Measurement speed

single scanning step ($\alpha_i, \Delta\beta_i$)	: $\approx 1 \text{ s}$
incomplete pole figure ($\alpha = 5^\circ - 85^\circ, \beta = 0^\circ - 360^\circ$)	: $\approx 20 \text{ min}$
ODF (4 pole figures, calculation)	: $\approx 1.5 \text{ h}$

HIGH TEMPERATURE EXPERIMENTS

With the equipment described above pole figures of a microalloyed low carbon steel were measured at different temperatures using MoK_α -radiation. At room temperature 3 incomplete BCC pole figures $\{110\}$, $\{200\}$ and $\{112\}$ were determined (Fig. 6). Subsequently the sample was automatically heated up to 950°C in the high temperature X-ray goniometer. During heating the ferrite to austenite transformation took place. The $\{111\}$, $\{200\}$ and $\{220\}$ FCC pole figures were determined at 950°C (Fig. 7).

The ODF's for both phases were calculated to investigate the texture transformation. Results of the *in situ* measurements and modelling of transformation textures are published elsewhere (Brückner, 1996a,b).

FIGURE 6 $\{110\}$, $\{200\}$ and $\{112\}$ pole figures of the ferritic phase of a recrystallized, low carbon steel at 20°C (MoK_α -radiation, $\alpha = 5^\circ\text{--}75^\circ$, $\beta = 0^\circ\text{--}360^\circ\text{C}$, measurement speed: ≈ 20 min/pole figure).

FIGURE 7 $\{111\}$, $\{200\}$ and $\{220\}$ pole figures of the austenitic phase of a low carbon steel at 950°C (MoK_α -radiation, $\alpha = 5^\circ\text{--}75^\circ$, $\beta = 0^\circ\text{--}360^\circ\text{C}$, measurement speed: ≈ 20 min/pole figure).

CONCLUSIONS

The design and construction of a hot stage for a four circle X-ray texture goniometer was described. This design allowed to determine pole figures at temperatures up to 1000°C and to obtain the ODF in about 25 min (measuring four quarter pole figures). The *in situ* measurements are designed to study texture evolution during heating to provide a deeper understanding of texture formation during phase transformations.

Acknowledgements

The financial support of the Deutsche Forschungsgemeinschaft is gratefully acknowledged.

References

- Bain, E.C. (1924) *Trans. AIME* **70**, 25–46.
Brückner, G., Aretz, W. and Gottstein, G. (1996a) submitted to *ICOTOM11*, Xi'an, China.
Brückner, G. and Pospiech, J. (1996b) submitted to *ICOTOM11*, Xi'an, China.
Hirsch, J., Burmeister, G., Hoenen, L. and Lücke, K. (1986) in *Experimental Techniques of Texture Analysis*, edited by H.J. Bunge, DGM Informationsgesellschaft Verlag, pp. 63–71.
Reher, F.R., Hänel, W., Czubayko, U. and Gottstein, G. (1994) *Mat. Sci. For.* **157–162**, 181–186.