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Neutron depolarisation study of the austenite grain size in TRIP steels

N.H. van Dijk[a,*], L. Zhao[b,c], M.Th. Rekveldt[a], H. Fredrikze[a], O. Tegus[d], E. Brück[d], J. Sietsma[b], S. van der Zwaag[e]

[a] *Interfaculty Reactor Institute, Delft University of Technology, Mekelweg 15, JB Delft 2629, The Netherlands*
[b] *TNW, TU Delft, Rotterdamseweg 137, 2628 AL Delft, The Netherlands*
[c] *NIMR, Rotterdamseweg 137, 2628 AL Delft, The Netherlands*
[d] *WZI, University of Amsterdam, Valcknierstraat 65, 1018 XE Amsterdam, The Netherlands*
[e] *Faculty of Aerospace Engineering, TU Delft, Kluyverweg 1, 2629 HS Delft, The Netherlands*

Abstract

We have performed combined neutron depolarisation and magnetisation measurements in order to obtain an in situ determination of the average grain size and volume fraction of the retained austenite phase in TRIP steels. The average grain size of the retained austenite was found to decrease for an increase in austenite volume fraction at two different annealing temperatures.

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Low-alloyed transformation-induced plasticity (TRIP) steels have recently attracted a growing interest for their high strength and good formability [1,2]. TRIP steels generally possess a multiphase microstructure containing ferrite (α-Fe), bainite, and (metastable) austenite (γ-Fe). The so-called TRIP effect in these steels arises from a martensitic transformation of the metastable retained austenite phase induced by external stress. It was found experimentally [3] and theoretically [4] that both the volume fraction and the grain size of the retained austenite play a crucial role in the TRIP properties as they significantly affect the mechanical stability of the retained austenite. In the present study we aim to determine the average grain size of the non-magnetic retained austenite within the ferromagnetic matrix by in situ neutron depolarisation measurements at room temperature. The advantage of this technique is that it probes a large volume in the bulk of the sample, which makes it relatively insensitive to a possible reduction in stability of the retained austenite at the surface.

The chemical composition of the studied $Al_{1.8}$ steel is 0.170 wt% C, 1.464 wt% Mn, 0.264 wt% Si, 1.805 wt% Al, 0.010 wt% P, and balance Fe. The

*Corresponding author. Tel.: +31-152786775; fax: +31-152788303.
E-mail address: nvijk@iri.tudelft.nl (N.H. van Dijk).

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as-received material was machined into sheets with dimensions of $50 \times 10 \times 1.0\,\mathrm{mm}^3$. The sheets were annealed for 10 min in an inert salt bath at an intercritical annealing temperature of either 800°C or 900°C, then quenched to 400°C in a salt bath, held at this temperature for 1 min, and subsequently quenched to room temperature in water. The applied heat treatment is chosen to obtain a multiphase microstructure with a maximum volume fraction of retained austenite and an absence of carbide precipitates [5]. In order to study the relation between the intercritical annealing temperature, where a two-phase mixture of ferrite and austenite is formed, and the retained austenite in the final microstructure two different annealing temperatures were studied. An increase in the intercritical annealing temperature is expected to result in an increase in the formed austenite fraction during annealing from $f_\gamma^{\mathrm{an}} = 0.25$ ($C_\gamma^{\mathrm{an}} = 0.59$ wt%) at $T_{\mathrm{an}} = 800°\mathrm{C}$ to $f_\gamma^{\mathrm{an}} = 0.38$ ($C_\gamma^{\mathrm{an}} = 0.40$ wt%) at $T_{\mathrm{an}} = 900°\mathrm{C}$.

In order to be able to analyse the neutron depolarisation data the volume fraction of retained austenite f_γ and the magnetic saturation induction of the ferromagnetic matrix (consisting of ferrite and bainite) $\mu_o M_s^\alpha$ were determined by magnetisation measurements performed on a Quantum Design SQUID magnetometer (MPMS-5S). The field-dependent magnetisation data (up to 5 T) from the as-received and heat-treated samples at room temperature were analysed within the framework developed in a previous experiment [5]. The derived magnetic saturation induction of the ferromagnetic matrix is $\mu_o M_s^\alpha = 2.043(2)\,\mathrm{T}$ and the volume fractions of retained austenite are $f_\gamma = 0.045(1)$ for $T_{\mathrm{an}} = 800°\mathrm{C}$ and $f_\gamma = 0.085(1)$ for $T_{\mathrm{an}} = 900°\mathrm{C}$. Surprisingly, in both cases about 20% of the initial austenite present at the intercritical annealing (f_γ^{an}) is found in the final microstructure as retained austenite (f_γ).

The neutron depolarisation experiments were performed on the ROG instrument at IRI. A thermal neutron beam was pulsed by a chopper with a pulse rate of 25 Hz and subsequently polarised (in the z-direction). The polarised neutron beam with a cross-section of $15 \times 3\,\mathrm{mm}^2$ was transmitted through the sample clamped between the poles of an electromagnet. The electromagnet provided an applied magnetic field up to 0.8 T oriented along the long axis of the sample and parallel to the polarisation of the incoming beam. The transmitted neutron beam was analysed by a polarisation analyser (oriented in the same direction as the polarisation of the incoming beam) and the resultant non-spin–flip intensity (I^{++}) was detected by a gas-filled ^{3}He detector. With an additional spin flipper between the polariser and the sample the polarisation of the transmitted beam was inverted and the spin–flip intensity (I^{+-}) was determined. The time of flight from the chopper to the detector allowed for wavelength-dependent measurements in the range of $\lambda = 1.2$–6 Å with a spread of $\Delta\lambda/\lambda = 2\%$. From the wavelength-dependent measurements of the spin–flip and non-spin–flip intensities the beam polarisation along the z-axis $P_z(\lambda) = (I^{++} - I^{+-})/(I^{++} + I^{+-})$ was determined. From two independent measurements of the beam polarisation in the absence of a TRIP sample, $P_z^\circ(\lambda)$, and in the presence of a TRIP sample, $P_z(\lambda)$, the polarisation transmission or depolarisation coefficient of the sample $D_{zz}(\lambda) = P_z(\lambda)/P_z^\circ(\lambda)$ is obtained.

The depolarisation coefficient $D_{zz}(\lambda)$ generally probes the spatial variation in the local magnetic induction $\mathrm{B}(x,y,z)$ oriented perpendicular to the polarisation P_z in the sample and can be expressed as [6]:

$$D_{zz}(\lambda) = \mathrm{e}^{-c\lambda^2 L(\alpha_{xx}+\alpha_{yy})}, \tag{1}$$

where $c = 2.15 \times 10^{29}\,\mathrm{T}^{-2}\,\mathrm{m}^{-4}$ is a constant, λ is the neutron wavelength, and L is the thickness of the sample (transmission length). The correlation parameters $\alpha_{ii}(i = x,y)$ contain the relevant information on the magnetic microstructure of the sample and can be expressed as

$$\alpha_{ii} = \left\langle \int_0^L \Delta B_i(x,y,z)\Delta B_i(x+x',y,z)\,\mathrm{d}x' \right\rangle, \tag{2}$$

where x is oriented along the polarised neutron beam, z along the beam polarisation, and $\langle \cdots \rangle$ refers to a spatial average over the sample volume illuminated by the cross-section of the polarised neutron beam. The variation in the local magnetic induction $\Delta\mathbf{B}(x,y,z)$ corresponds to the difference between the local magnetic induction $\mathbf{B}(x,y,z)$ and

its average value of $\langle \mathbf{B}(x,y,z) \rangle$ within the illuminated sample volume.

For isolated non-magnetic spherical austenite grains in a magnetically saturated ferromagnetic matrix $\alpha_{xx}+\alpha_{yy}$ can be expressed as [6]

$$\alpha_{xx} + \alpha_{yy} = \tfrac{3}{32} f_{\mathrm{m}}(1 - f_{\mathrm{m}}) B_{\mathrm{s}}^2 R, \qquad (3)$$

where $f_{\mathrm{m}} = f_{\alpha} = 1 - f_{\gamma}$ is the magnetic volume fraction of the ferromagnetic matrix, $B_{\mathrm{s}} = \mu_{\mathrm{o}} M_{\mathrm{s}}^{\alpha}$ is the magnetic saturation induction of the ferromagnetic matrix, and $R = R_{\gamma}$ is the average radius of the non-magnetic austenite grains. In the present study we have determined the magnetic volume fraction and the saturation induction of the ferromagnetic matrix from magnetisation measurements. Based on these results and the above equations it is possible to extract the average grain size from the experimental depolarisation coefficient $D_{zz}(\lambda)$ if the sample is close to magnetic saturation. When the applied magnetic field is not sufficiently strong to approach saturation magnetic domains are formed in the ferromagnetic matrix, which are an additional source of depolarisation.

In Fig. 1 the experimental depolarisation coefficient $D_{zz}(\lambda)$ for the $Al_{1.8}$ TRIP steel annealed at $T_{\mathrm{an}} = 900°C$ is shown as a function of the squared neutron wavelength λ^2 in applied magnetic fields up to 0.8 T. The data in Fig. 1 confirm the predicted proportionality between $\ln(D_{zz})$ and λ^2. In Fig. 2 the fitted correlation parameter $\alpha_{xx}+\alpha_{yy}$ is shown as a function of the applied magnetic field. For $\mu_{\mathrm{o}}H > 0.6$ T the value of $\alpha_{xx}+\alpha_{yy}$ approaches saturation as the magnetic domains are gradually oriented towards the applied magnetic field. At the maximum applied magnetic field of $\mu_{\mathrm{o}}H = 0.8$ T the model of Eq. (3) can be used to determine the average diameter $D_{\gamma} = 2R_{\gamma}$ of the non-magnetic austenite grains in the ferromagnetic matrix. The deduced austenite grain diameter for the $Al_{1.8}$ TRIP steels decreases by roughly 50% for the increase in annealing temperature, from $D_{\gamma} = 4.8(1)\,\mu$m for $T_{\mathrm{an}} = 800°C$ to $D_{\gamma} = 2.19(4)\,\mu$m for $T_{\mathrm{an}} = 900°C$. The corresponding volume fraction of retained austenite is roughly doubled for the increase in annealing temperature from $f_{\gamma} = 0.045(1)$ for $T_{\mathrm{an}} = 800°C$ to $f_{\gamma} = 0.085(1)$ for $T_{\mathrm{an}} = 900°C$. The inverse correlation between f_{γ} and D_{γ} is possibly caused by the stability of the larger austenite grains being reduced for an increasing austenite fraction due to the lower average carbon concentration in the retained austenite.

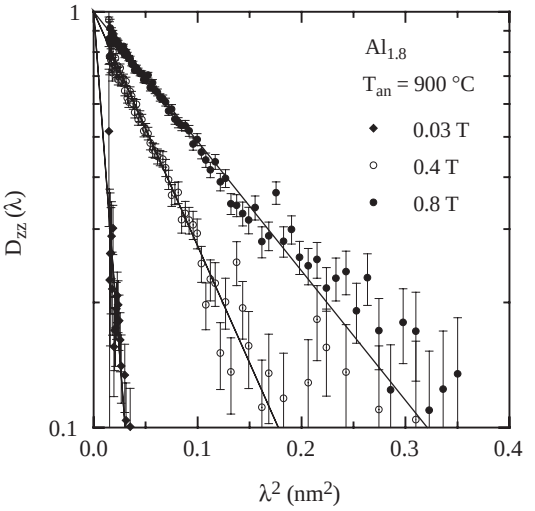

Fig. 1. Experimental depolarisation coefficient $D_{zz}(\lambda)$ as a function of the neutron wavelength squared λ^2 for the $Al_{1.8}$ TRIP steel annealed at a temperature of $T_{\mathrm{an}} = 900°C$ in different applied magnetic fields up to 0.8 T.

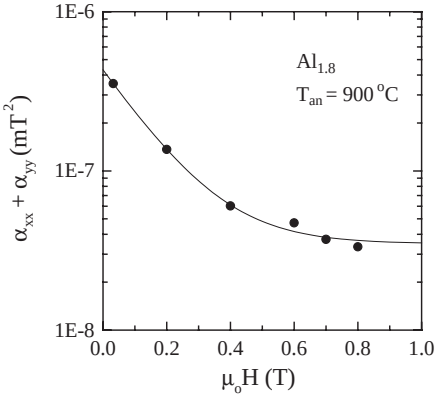

Fig. 2. Variation of $\alpha_{xx}+\alpha_{yy}$ as a function of the applied magnetic field $\mu_{\mathrm{o}}H$ for the $Al_{1.8}$ TRIP steel annealed at a temperature of $T_{\mathrm{an}} = 900°C$. The solid line is a guide to the eye.

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