Quantification of TRIP Kinetics in Medium Mn Steels by in-situ Magnetic Measurements

Michael Callahan, Olivier Hubert, Jean-Hubert Schmitt

Quantification of TRIP Kinetics in Medium Mn Steels by in-situ Magnetic Measurements

CALLAHAN Michael[1], HUBERT Olivier[2], SCHMITT Jean-Hubert[1],

[1]MSSMat, CNRS, CentraleSupélec, Univerité Paris-Saclay, 92290 Châtenay-Malabry, France

[2]LMT-Cachan, CNRS UMR 8535, ENS Cachan, Université Paris-Saclay, 94235 Cachan Cedex, France

Abstract: Increasing interest of the automotive industry in light, high strength – high formability steels has led to the development of a third generation of advanced high strength steels (AHSS). So-called Medium-Mn alloys belong to this generation of steel. Medium-Mn alloys are ultra-fine-grained dual phase steels containing a mixture of ferrite and retained austenite. The stability of the retained austenite controls the degree to which transformation induced plasticity (TRIP) or twinning induced plasticity (TWIP) may be observed. In the current study Medium-Mn alloys of varying degrees of austenite stability are studied, all of which exhibit the TRIP deformation mechanism. The kinetics of the phase transformation are evaluated by using in-situ measurements of the magnetic properties of a sample to determine the volume fraction of retained austenite as a function of the plastic strain during tensile testing. Results are compared to phase volume fraction measurements made by X-ray diffraction (XRD). The magnetic measurement is shown to be as robust as XRD while providing a much richer data set with a shorter overall experiment duration.

Key words: third generation steels, AHSS, medium Mn, TRIP, TWIP, magnetic susceptibility, Lüders bands

1 Introduction

Efforts of the automotive industry to reduce carbon emissions of their vehicles has led to the development of a third generation of advanced high strength steel (AHSS) called “Medium-Mn” steels. Medium-Mn steels are capable of displaying transformation-induced plasticity (TRIP) or twinning-induced plasticity (TWIP) depending on the stability of the retained austenite. By varying the annealing temperature used (within the intercritical ferrite+austenite region), one can change the stability of the austenite phase and make it more or less susceptible to either TRIP or TWIP [1-4]. While it is possible to simulate the microstructure that should be obtained for a certain composition and thermal treatment schedule [5,6], it is more difficult to accurately predict the mechanical properties of the steel due to the highly localized stresses/strains that can occur with TRIP and TWIP. It is thus of interest to understand the kinetics of the TRIP phenomenon as a function of strain if one wishes to properly simulate the mechanical behavior for a given microstructure or to correlate TRIP to the stability of the austenite via its stacking fault energy (SFE). Conveniently, the transformation of austenite into martensite during TRIP is a transformation from a paramagnetic phase to a ferromagnetic one. As such, measurements of the saturation magnetization using commercial ferritescopes are sometimes used to study martensite transformation in TRIP steels or shape memory alloys. This research seeks to employ an improved methodology using the intrinsic magnetic properties of the material.

2 Materials and Methods

2.1 Material Fabrication

In order to vary the microstructure and more importantly the austenite stability of the samples to be studied, sheets of Medium-Mn TRIP steel were hot-rolled, quenched, and intercritically annealed at three different intercritical annealing temperatures T_{IA}: 740°C, 760°C, and 780°C. Flat tensile samples were then cut from the resulting sheets and unidirectional tensile tests were performed using digital image correlation (DIC) to study strain heterogeneities. Tensile tests were performed at a strain rate of $5x10^{-4}s^{-1}$. The composition of the steel is provided in Table 1.

Table 1 Composition of Medium-Mn TRIP steel (wt %)

Fe	C	Mn	Al
bal.	0.2	5.1	2.5

2.2 Magnetic Measurement Methodology

The measurement of the volume fraction of ferrite + martensite—both ferromagnetic phases—via magnetism typically utilizes the saturation value of the sample magnetization obtained for a sufficiently strong applied magnetic field. This method of measurement allows a rapid acquisition speed as the applied magnetic field can be rapidly cycled. The ferromagnetic volume fraction in a Medium Mn TRIP steel can then be calculated using a simple mixture rule [7],

$$M^s = f_{\alpha+\alpha'}\, M_\alpha^s + f_\gamma\, M_\gamma^s \quad (1)$$

where M^s is the instantaneous saturation magnetization, f_i gives the volume fraction of phase *i* and M_i^s is the saturation magnetization of phase *i (the ferromagnetic phase is denoted a+a' since both pro-eutectoid ferrite a and martente a' are ferromagnetic and cannot be separated)*. In the current case, M_γ^s is taken as 0 because the magnetic response of the paramagnetic austenite is negligible compared to that of the ferromagnetic ferrite or martensite. Equation 1 is thus simplified to

$$f_{\alpha+\alpha'} = {M^s}/{M_\alpha^s} \quad (2).$$

This method, referred to hereafter as the hysteretic magnetic measurement, has the advantage of a high sampling rate. However, it suffers from inaccuracy at high retained austenite volume fractions (≥ ~0.2) because it assumes that the magnetic field is homogeneous throughout the microstructure.

An alternative method of measuring the phase volume fractions through magnetic measurements is to perform an anhysteretic measurement from which the sample's magnetic susceptibility can be obtained [8]. Applying Equation 2 for a given magnetic field H, one obtains

$$f_{\alpha+\alpha'} = \left|\frac{M}{H}\cdot\left(\frac{M_{\alpha+\alpha'}}{H}\right)^{-1}\right| = \left|\frac{M}{H}\cdot\left(\frac{M_{\alpha+\alpha'}}{H_{\alpha+\alpha'}}\right)^{-1}\cdot\frac{H}{H_{\alpha+\alpha'}}\right| \quad (3)$$

which can be simplified to

$$f_{\alpha+\alpha'} = \frac{\chi_{\varepsilon^p}}{\chi_{\alpha+\alpha'}}\frac{H}{H_{\alpha+\alpha'}} \quad (4)$$

Where, $\chi_{\varepsilon^p} = (M/H)$ is the susceptibility measured for a sample deformed to a plastic strain of ε^p and χ_α is the susceptibility of a 100% ferromagnetic material. The susceptibility $\chi_\alpha = 1.4\text{x}10^6$ A/m was measured by anhysteretic measurements of a sample quenched in liquid nitrogen to fully transform the retained austenite.

Finally, the magnetic field H is localized using an Eshelby problem for a spherical grain of austenite or ferrite/martensite in a homogeneous equivalent medium [9, 10]:

$$f_{\alpha+\alpha'} = \frac{X_{\varepsilon^p}\,(3+2X_{\varepsilon^p}+X_\alpha)}{X_\alpha(3+3X_{\varepsilon^p})} \quad (5)$$

2.3 *In-Situ* Magnetic Measurement Setup

A conducting coil of 50 loops was wound around the surface of the sample over about 1cm of the gauge length. This "secondary" coil served to measure the magnetic response of the sample when submitted to a magnetic field imposed by a "primary" coil placed around the sample. The sample/coil system is placed between two insulating pieces which is able to be mounted on the tensile machine.

In-situ magnetic measurements were made during tensile tests on samples of each of the 3 TRIP alloys studied in order to determine the effect of the intercritical annealing temperature on the TRIP kinetics. The magnetic field was cycled between $\pm1.5\text{x}10^4$ A/m at a frequency of 2Hz during tensile testing.

3 Results and Discussion

3.1 Tensile Results

Unidirectional tensile testing using an extensometer (12.5 mm gauge length versus 60mm active tensile length of the sample) revealed a stepwise plastic deformation behavior in samples annealed at T_{IA}=740°C and 760°C, as seen in Figure 1. An intial yield point elongation indicates the presence of a Lüders band propagating along the tensile length of the sample. No such strain localizations were observed in the samples annealed at T_{IA}=780°C.

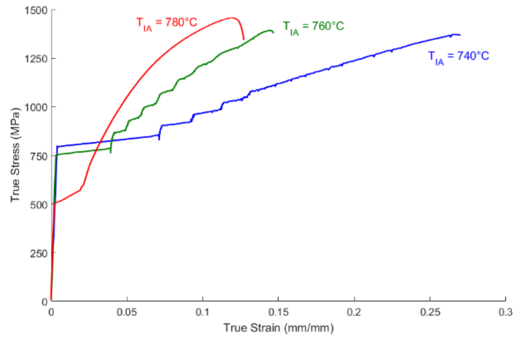

Figure 1 – Representative unidirectional tensile data for each of the intercritical annealing temperatures used.

Digital image correlation data confirmed the presence of the Lüders band, shown in Figure 2 for a sample annealed at T_{IA}=740°C. Once the Lüders band had fully traversed the sample, several subsequent bands propagated along the sample one after the other. This highly heterogeneous plasticity mechanism continued until necking and rupture. The instantaneous velocity of the strain bands were calculated and shown to decrease with each subsequent band.

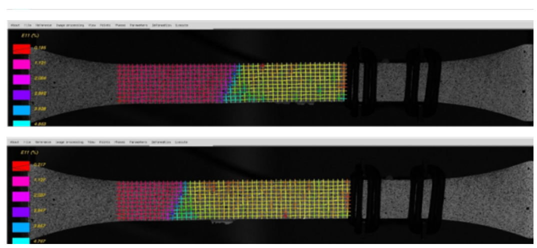

Figure 2 - DIC results for a sample annealed at 740°C show the passage of deformation bands. The left end of the sample is fixed and the right is mobile. The yellow zone corresponds to a strain of approximately 8% and the pink zone is still elastic at this point. The black bands are rubber bands used to fix an extensometer to the opposite side of the sample.

3.2 Magnetic Measurement Validation

Measurements of the volume fraction of ferrite+martensite (referred to in the following as the ferromagnetic volume fraction) made by measurements of the magnetic susceptibility and saturation were compared to measurements made by X-ray diffraction (XRD), as shown in Figure 3. The magnetic susceptibility measurements were in good agreement with XRD results. The saturation method, while it seemed to follow a believable trend, is shifted towards lower values of the ferromagnetic volume fraction. This reinforces the need to initialize the saturation measurements with measurements made via the magnetic susceptibility.

A significant effect of the stress on the saturation magnetization was observed during unloading of the sample annealed at T_{IA}=760°C. This could account for some of the error observed in the volume fractions obtained via the saturation magnetization and requires correction. Figure 5 shows magnetic hystereses for several levels of stress during unloading; the saturation magnetization increased by about 25% of the saturation value at 1000MPa.

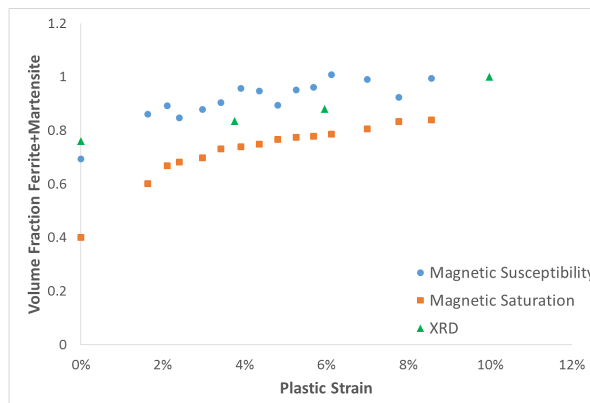

Figure 3 - Comparison of ferromagnetic volume fractions measured by magnetic susceptibility, magnetic saturation, and X-ray diffraction for a sample annealed at T_{IA}=780°C.

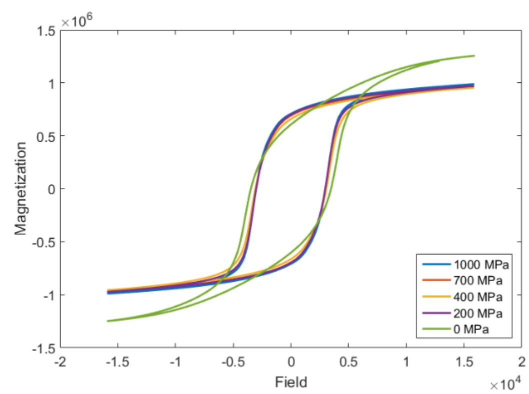

Figure 4 - Magnetization-Field hystereses during unloading of the sample annealed at 760°C. It can be seen that the sample magnetization increases as the load is removed, indiating a magneto-mechanical coupling that must be corrected.

3.3 In-Situ Results

In-situ experiments were performed for samples annealed at T_{IA}=740°C, 760°C, and 780°C. Figure 5 plots the true stress (in black) and the ferromagnetic volume fraction (in blue) versus the true plastic strain for each of the three experiments. It can be seen that for T_{IA}=760°C and 780°C there was a sharp increase in the ferromagnetic volume fraction within the first few percent of plastic strain, after which the transformation rate gradually slowed down. For the sample annealed at 740°C, the measured volume fraction seems relatively constant; however, this is due to the fact that the applied load causes the magnetization measured to decrease with respect to a measurement made without an applied charge.

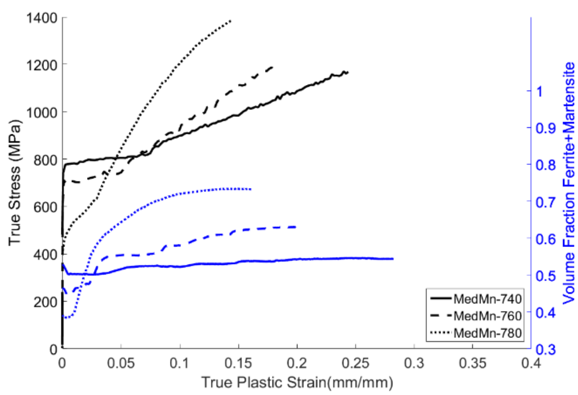

Figure 5 - *In-situ* test data for samples annealed at 740°C, 760°C, and 780°C. The true stress (black) and ferromagnetic volume fraction (blue) are plotted as a function of the true plastic strain.

It was observed that a stepwise behavior is present in both the tensile and magnetic data for samples annealed at 740°C and 760°C. The steps in tensile curves were determined to be the result of Lüders-type deformation bands; thus, it seems that the passage of these bands and the martensitic transformation coincide with one another. It is suspected that the localization of the plastic strain ahead of the strain band induces the martensite transformation; however, the sequence of events as the strain band passes through a point in the sample has not yet been determined.

4 Conclusions

Measurements of the magnetic properties of tensile samples were made in-situ in order to quantify the volume fraction of the ferrite+martensite phases (the ferromagnetic phases) as a means of characterizing the kinetics of the TRIP phenomenon in this alloy for various levels of retained austenite stability. The results showed that magnetic measurements of the phase volume fractions were in strong agreement with XRD measurements. In-situ measurements provided volume fraction measurements at a frequency of 2Hz, leading to a much richer data set than is feasible using XRD. The austenite to martensite transformation was correlated to an increase in work hardening just after yielding and to the passage of deformation bands observed in tension. The method does however require a correction of the effect of the applied stress on the saturation magnetization. This small complication aside, the magnetic method appears to be very promising for study of TRIP behaviors, especially when large data set sizes are desired.

5 Acknowledgements

The authors would like to thank Mrs. Astrid Perlade and Mr. Ian Zuazo of ArcelorMittal for providing the material. This study has been performed within the framework of the ANR 13-RMNP-0002 MedMnAl Steels founded by the Agence Nationale de la Recherche (ANR). This project is also supported by the competitive cluster "Materalia".

References

[1] A. Arlazarov, M. Gouné, et al., "Evolution of microstructure and mechanical properties of medium Mn steels during double annealing," Mater. Sci. Eng. A, vol. 542, pp. 31–39, Apr. 2012.

[2] Z. H. Cai, H. Ding, et al., "Austenite stability and deformation behavior in a cold-rolled transformation-induced plasticity steel with medium manganese content," Acta Mater., vol. 84, pp. 229–236, 2015.

[3] P. J. Gibbs, E. De Moor, et al., "Austenite stability effects on tensile behavior of manganese-enriched- austenite transformation-induced plasticity steel," Metall. Mater. Trans. A Phys. Metall. Mater. Sci., vol. 42, no. 12, pp. 3691–3702, Apr. 2011.

[4] S. Lee and B. C. De Cooman, "Tensile Behavior of Intercritically Annealed Ultra-Fine Grained 8% Mn Multi-Phase Steel," Steel Res. Int., vol. 86, no. 10, pp. 1170–1178, 2015.

[5] H. J. Lee, S. S. Sohn, S. Lee, J. H. Kwak, and B. J. Lee, "Thermodynamic analysis of the effect of C, Mn and Al on microstructural evolution of lightweight steels," Scr. Mater., vol. 68, no. 6, pp. 339–342, Mar. 2013.

[6] R. Wei, M. Enomoto, R. Hadian, H. S. Zurob, and G. R. Purdy, “Growth of austenite from as-quenched martensite during intercritical annealing in an Fe-0.1C-3Mn-1.5Si alloy,” Acta Mater., vol. 61, no. 2, pp. 697–707, Jan. 2013.

[7] F. Mballa-Mballa, O. Hubert, S. Lazreg, and P. Meilland, “Multidomain modelling of the magneto-mechanical behaviour of dual- phase steels,” 8th World Conference on Nondestructive Testing, Apr 2012, Durban, South Africa, pp. 16–20, 2012.

[8] C. Bormio-Nunes and O. Hubert, “Piezomagnetic behavior of Fe–Al–B alloys,” J. Magn. Magn. Mater., vol. 393, pp. 404–418, 2015.

[9] L. Daniel, O. Hubert, N. Buiron, and R. Billardon, “Reversible magneto-elastic behavior: A multiscale approach,” J. Mech. Phys. Solids, vol. 56, no. 3, pp. 1018–1042, 2008.

[10] J. D. Eshelby, “The determination of the elastic field of an ellipsoidal inclusion, and related problems.” proceedings of the royal society, 1957.