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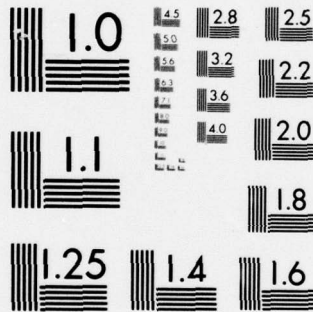
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# AC-PERMEABILITY MEASUREMENT OF STRAIN-INDUCED MARTENSITE DURING TENSILE DEFORMATION OF TRIP STEELS

GREGORY B. OLSON, MORRIS AZRIN, and HANS E. BAND  
METALS RESEARCH DIVISION

March 1977



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OF TRIP STEELS

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ABSTRACT

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An AC magnetic permeability method has been developed for continuously monitoring the course of the strain-induced martensitic transformation during tensile deformation of TRIP steels. True stress-strain and magnetic measurements were made simultaneously during both inhomogeneous deformation (Lüders-band formation) and uniform flow. Calibration with quantitative optical metallography and density measurements indicates that the AC-permeability technique yields accurate curves for the volume fraction martensite versus plastic strain. The transformation curve in high-strength TRIP steel has a sigmoidal shape consistent with that reported for lower strength austenitic steels.

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## INTRODUCTION

High-strength metastable austenitic steels known as TRIP steels<sup>1</sup> exhibit unusually high ductility and toughness as a result of a strain-induced martensitic transformation in service. The mechanical behavior of these materials is complicated by the formation of Lüders bands upon initial yielding. Only recently have true stress and true strain measurements been made which incorporate this regime of inhomogeneous flow.<sup>2</sup> These studies have called attention to the unusual shape of the stress-strain curve and the importance of the temperature dependence of the transformation kinetics in causing a high apparent strain-rate sensitivity of the flow properties. Accordingly, a better understanding of the mechanical behavior of TRIP steels requires a determination of the precise shape of the strain-induced transformation curve (volume fraction martensite versus plastic strain) as well as the temperature dependence of this shape.

Measurements of transformation curves in lower strength metastable austenites have produced sigmoidal-shaped curves,<sup>3-5</sup> a shape which has been predicted by theory of strain-induced nucleation.<sup>6</sup> Gerberich et al.<sup>7</sup> have proposed a parabolic relation for the transformation curve in high-strength TRIP steels. Although a significant difference between sigmoidal and parabolic shapes exists at low strains, this difference is equivocal in the case of TRIP steels due to the lack of accurate martensite volume fraction versus true strain data during the initial inhomogeneous flow associated with yielding (Lüders-band formation). In applying the theoretical concepts developed from lower strength alloys, it is important to establish any fundamental differences in transformation behavior between those alloys and TRIP steels. This study was undertaken to determine the true shape of the transformation curve in high-strength TRIP steels and to establish a convenient method for monitoring the change of curve shape with temperature.

## MAGNETIC TECHNIQUE AND INSTRUMENTATION

When the austenitic phase is paramagnetic, as in the alloys considered here, the formation of ferromagnetic martensite can be conveniently monitored by magnetic techniques. The underlying assumption of such approaches is that all the other constituents are nonferromagnetic and that their magnetic effects can be neglected. The magnetic susceptibility should then vary linearly with the volume fraction of the ferromagnetic constituent (i.e., martensite). Several DC magnetic

1. ZACKAY, V. F., PARKER, E. R., FAHR, D., and BUSCH, R. *The Enhancement of Ductility in High-Strength Steels*. Trans. ASM, v. 60, 1967, p. 252-259.
2. AZRIN, M., OLSON, G. B., and GAGNE, R. A. *Inhomogeneous Deformation and Strain-Rate Effects in High-Strength TRIP Steels*. Army Materials and Mechanics Research Center, AMMRC TR 73-12, March 1973; also Mat. Sci. and Eng., v. 23, no. 1, April 1976, p. 33-41.
3. ANGEL, T. *Formation of Martensite in Austenitic Stainless Steels*. J. Iron and Steel Inst., v. 177, 1954, p. 165-174.
4. POWELL, G. W., MARSHALL, E. R., and BACKOFEN, W. A. *Strain Hardening of Austenitic Stainless Steel*. Trans. ASM, v. 50, 1958, p. 479-497.
5. LECROISEY, F., and PINEAU, A. *Martensitic Transformations Induced by Plastic Deformation in the Fe-Ni-Cr-C System*. Met. Trans., v. 3, 1972, p. 387-396.
6. OLSON, G. B., and COHEN, M. *Kinetics of Strain-Induced Martensitic Nucleation*. Met. Trans., v. 6A, 1975, p. 791-795.
7. GERBERICH, W. W., HEMMINGS, P. L., ZACKAY, V. F., and PARKER, E. R. *Interactions Between Crack Growth and Strain-Induced Transformation*. Fracture 1969, ed. P. L. Pratt, Chapman and Hall Ltd., London, 1969, p. 288-305.

techniques can be employed based on saturation magnetization,<sup>8</sup> Hall effect,<sup>9</sup> mechanical force (e.g., "Magne-Gage"),<sup>3</sup> Barkhausen effect,<sup>10</sup> magneto-resistivity (suitable mostly for low temperatures), or hysteresis loop evaluation. Of these, the most accurate method applied to alloys of this type is the measurement of saturation magnetization in a high magnetic field.<sup>5,8</sup> The sample is first magnetized to saturation with a DC magnetizing coil; then the current is reversed to produce saturation in the opposite direction, and the resultant flux change is determined with a suitable integrating flux meter. After subtracting the air flux density ( $\mu_0 H_{\max}$ ), the saturation induction is found from the geometry of the sample and the measured flux change. This method has the advantage that the saturation magnetization of the sample is linearly related to the volume fraction of the ferromagnetic phase, and is not significantly influenced by elastic and plastic strains. However, because high fields are needed to reach saturation, mechanical testing must be interrupted to move the specimen to a separate magnetic measurement fixture,<sup>8</sup> or if the measurements are made continuously during testing, the transformation behavior may be influenced by the increased thermodynamic driving force associated with the magnetic field.<sup>11</sup>

Ferromagnetic content can be monitored at very low magnetic fields using techniques sensitive to the initial magnetic permeability. Methods based on AC permeability rely on the change in the impedance of a sensing coil whose flux path is partially filled by the specimen. The inductive-reactance component of the coil impedance responds to changes of the effective AC permeability of the specimen as its ferromagnetic content increases. The permeability change can be sensed by measurement of either voltage ("transformer" method) or frequency ("resonant circuit" method). One such transformer technique, which has been applied with limited success to TRIP steels,<sup>12</sup> is to use the sensing coil surrounding the specimen as the secondary winding of a transformer, and to measure the magnitude and phase of the induced AC voltage when the primary winding is driven by a known AC reference voltage. To facilitate continuous measurement during mechanical testing, we chose a technique based on AC permeability, using a resonant circuit method in which the inductive component of the coil impedance is determined by resonating it with a known capacitance and measuring the frequency ( $\nu$ ) of the signal. An important advantage of this method is the nondependence of the output frequency on oscillator voltage, thereby eliminating drift and coil-alignment errors usually associated with other types of AC-permeability methods. Another potential advantage relative to voltage measurement is that signal conditioning of the oscillator voltage could be used before measuring its frequency (i.e., noise reduction and wave shaping). Though convenient for continuous measurement, the measured parameter is not as simply related to the material properties as in the saturation-magnetization method. Hence, extensive calibration by other techniques is required.

8. DeMIRAMON, B. *Quantitative Investigation of Strain-Induced Strengthening in Steel*. Univ. of Calif., Berkeley, UCRL-17849, September 1967.
9. FICKETT, F. R. *Martensite Transformation Detection in Cryogenic Steels (Magnetometer Development)*. NBS Technical Note G13, December 1971.
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11. KORENKO, M. K. *Martensitic Transformations in High Magnetic Fields*. Sc. D. Thesis, Mass. Institute of Tech., Department of Metallurgy and Materials Science, Cambridge, MA, February 1973.
12. MAXWELL, P. C., SHYNE, J. C., and GOLDBERG, A. *TRIP Effect in Fe-Ni-C Alloys*. Univ. of Calif., Livermore, UCRL-51215, April 1972.

The basic geometry of the test specimen and the coil surrounding the gage section is represented schematically in Figure 1. The specimen was mounted in the testing machine using nonmagnetic spacers, pins, and grips (clevis type) in contact with the specimen grip ends, in order to interrupt the magnetic circuit to the ferromagnetic parts of the machine. This causes the flux linking the sensing coil to take an air return path just outside the specimen gage section, rather than pass through the machine. The only magnetic material thus linked by the flux is the transformed portion of the specimen gage length. The sensing coil constitutes one of the reactive elements of an LC resonant circuit which is the frequency-determining part of a transistorized audio oscillator. A schematic diagram of the audio oscillator circuit is shown in Figure 2. The basic circuit is that of a Colpitts two-terminal oscillator using a 2N2907A type PNP silicon transistor. Power was supplied to the oscillator at 22 volts DC from a dry cell. A bias-control potentiometer is provided in the base circuit.

The total instrumentation used for the magnetic measurement is represented schematically in Figure 3. The oscillator frequency was read by a Hewlett-Packard analog frequency meter. For chart recording of the frequency, the single-ended DC output of the analog frequency meter was converted to the double-ended input of an oscillograph system using a Keithley Differential Amplifier with a dummy DC voltage source at one of its input channels. This arrangement provided a suitable match between the frequency meter output and the oscillograph input, as well as additional gain. The final output was obtained from a Sanborn multichannel strip chart recorder which simultaneously recorded the mechanical test data. For added precision the oscillator frequency was also read visually from a digital frequency meter and recorded periodically on the Sanborn chart by hand.

### SENSING COIL DESIGN

A major objective of this program was to obtain volume fraction martensite versus true strain measurements during the initial inhomogeneous flow upon yielding (Lüders-band formation) in high-strength TRIP steels. It is not feasible to

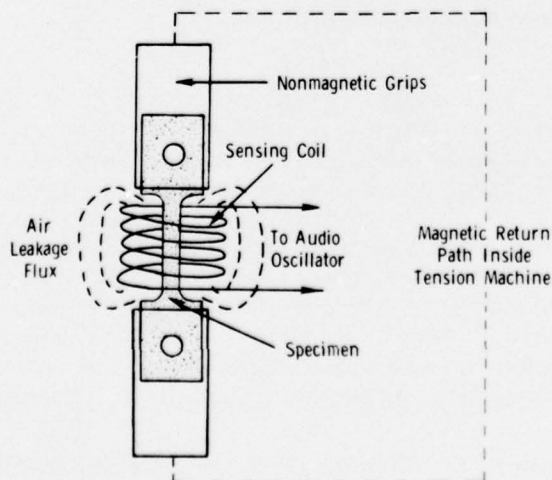


Figure 1. Sensing coil, magnetic flux, and specimen geometry.

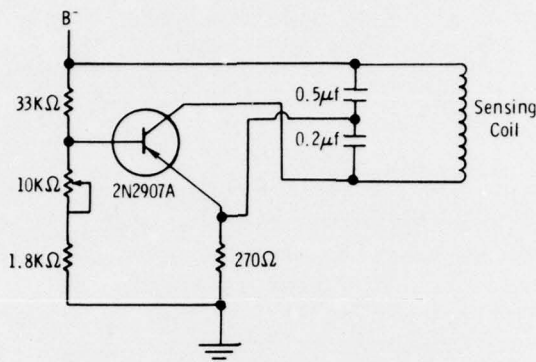


Figure 2. Schematic diagram of transistorized audio oscillator circuit.

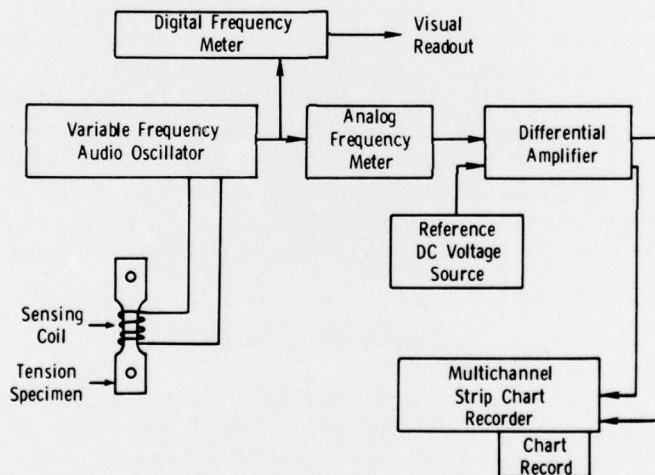


Figure 3. Instrumentation for magnetic measurements.

confine the magnetic flux sufficiently to obtain a local measurement of ferromagnetic volume fraction at the peak strain position (minimum cross-sectional area) in a forming Lüders band without considerable averaging over the surrounding lower strain portions of the band. Accurate local strain measurements, however, can be made continuously during a tension test.<sup>2,13</sup> Accordingly, it was decided to design the sensing coil in such a way that the *total* martensite content of the specimen gage section could be monitored during continuous measurement of the entire specimen strain profile. If the volume fraction of strain-induced martensite ( $f$ ) is a single-valued function of true tensile strain ( $\epsilon$ ), this is all the information required to uniquely determine  $f$  versus  $\epsilon$  during the inhomogeneous flow. The data analysis procedure adopted for this purpose is described in a later section.

13. NUNES, J., and LARSON, F. R. *A Method for Determining the Plastic Flow Properties of Sheet and Round Tensile Specimens.* Army Materials and Mechanics Research Center, WAL TR 111.1/1, March 1961.

Flat tension specimens were used with a 1-inch (2.54-cm) gage section, 0.20-inch (0.508-cm) width, and 0.10-inch (0.254-cm) thickness. In order to detect the total martensite content in this volume, a rectangular cross-section coil was made 1.25 inches (3.18 cm) in length to completely surround the gage section. The coil interior dimension of 0.20 inch (0.508 cm) in the specimen thickness direction was as close to the specimen as practical for high sensitivity. The interior dimension of 1.25 inches (3.18 cm) in the specimen width direction was just wide enough to provide clearance for continuous measurement of the specimen width profile.

The coil was attached to an adjustable stainless steel track which could slide along the specimen grips. It was held in position by springs such that the coil was always centered between the grips. Paramagnetic materials were used for the grips, track, and springs so as to exert a minimal influence on the coil frequency.

The coil itself was constructed from 500 turns of No. 22 copper wire bonded by Sauereisen cement suitable for the desired temperature range of -196 C to 200 C. The coil resting frequency, or the frequency with an air core in the coil, was approximately 16 kHz. Placing a fully paramagnetic specimen in the coil produced a negligible frequency increase on the order of 2 Hz.

#### COIL CALIBRATION

The TRIP steel used in this study was of nominal composition Fe-9Cr-8Ni-4Mo-2Si-0.8Mn-0.27C, corresponding to the A-3 alloy studied by Zackay et al.<sup>1</sup> The alloy was air melted and poured under an argon atmosphere, hot worked at 1150 C, and homogenized at 1250 C. The material was examined in both the solution-treated (1200 C, 1 hr) and warm-rolled (80% reduction at 450 C) conditions. For calibration purposes it was desired to produce various amounts of spontaneous martensitic transformation by cooling to low temperatures, since the coarse plate martensite formed in this manner is ideally suited for quantitative metallography. However, the solution-treated alloy exhibited only a small amount of spontaneous transformation after isothermal holding at -196 C for several hours. Therefore, a portion of the material was decarburized to 0.19 carbon after which significant amounts of spontaneous transformation could be produced at temperatures below -100 C. Samples of the decarburized material were machined to the dimensions of the tension specimen gage sections (1.0 x 0.20 x 0.10 inch or 2.54 x 0.508 x 0.254 cm) and cooled to various temperatures to produce different amounts of martensite. The frequency decrease upon placing them in the coil was measured, and the martensite contents were determined independently with a Quantimet optical analyzer.

The magnitude of the frequency decrease  $|\Delta\nu|$  is plotted versus martensite volume fraction in Figure 4. The dependence is reasonably linear, and the coil sensitivity,  $S = -d\nu/df$ , is approximately 20 Hz per 1% martensite.

In order to evaluate the dependence of coil sensitivity on the specimen shape during deformation, constant volume standards representing the shape of the specimen gage section at various elongations were machined from a ferromagnetic

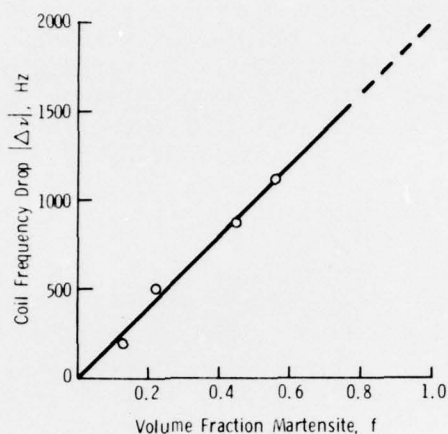


Figure 4. Magnitude of coil frequency change  $|\Delta\nu|$  versus martensite volume fraction  $f$  for calibration standards produced by spontaneous transformation on cooling.

1018 steel. Measuring the frequency drop for each standard, it was found that the coil sensitivity was constant within 2% out to an equivalent elongation of 50%. Since elongations encountered during testing were within this range, no correction was applied for specimen shape.

The coil resting frequency, which was approximately 16 kHz at room temperature, increased slightly as temperature was lowered down to -150 C. Below -150 C, the frequency increased more rapidly, reaching about 17 kHz at liquid nitrogen temperature (-196 C). Comparing the resting frequency with the frequency obtained on cooling with a ferromagnetic standard in the coil, the coil sensitivity was found to be constant within 0.6% down to -125 C and increased by 10% at -196 C.

The paramagnetic specimen grips exerted some influence on the coil resting frequency. To correct for this, the resting frequency was determined as a function of crosshead displacement. The gage section was removed from a tension specimen and the resting frequency versus crosshead displacement was measured with the specimen grip ends positioned in the grips in order to more accurately simulate the conditions during a test. Frequency drops during actual runs were determined relative to this changing resting frequency. The correction amounted to only a few hertz and was of significance only when detecting small amounts of martensite formation at yielding.

#### STRESS AND STRAIN MEASUREMENTS

The mechanical test apparatus was essentially that described in References 2 and 13. A 30,000-lb (130,000 N) capacity Tinius Olsen testing machine was used with crosshead motion (0.02 inch/min, 0.05 cm/min) monitored by a deflectometer to obtain load-displacement curves. To obtain true-strain measurements during inhomogeneous flow, the specimen width profile was monitored by "fingers" which

continuously traversed the gage section. The transverse displacement of these fingers was measured by strain gages mounted on cantilever arms to which the fingers were attached as illustrated in Figure 5. In order not to influence the coil resting frequency, the fingers were constructed from nonconductive glass-

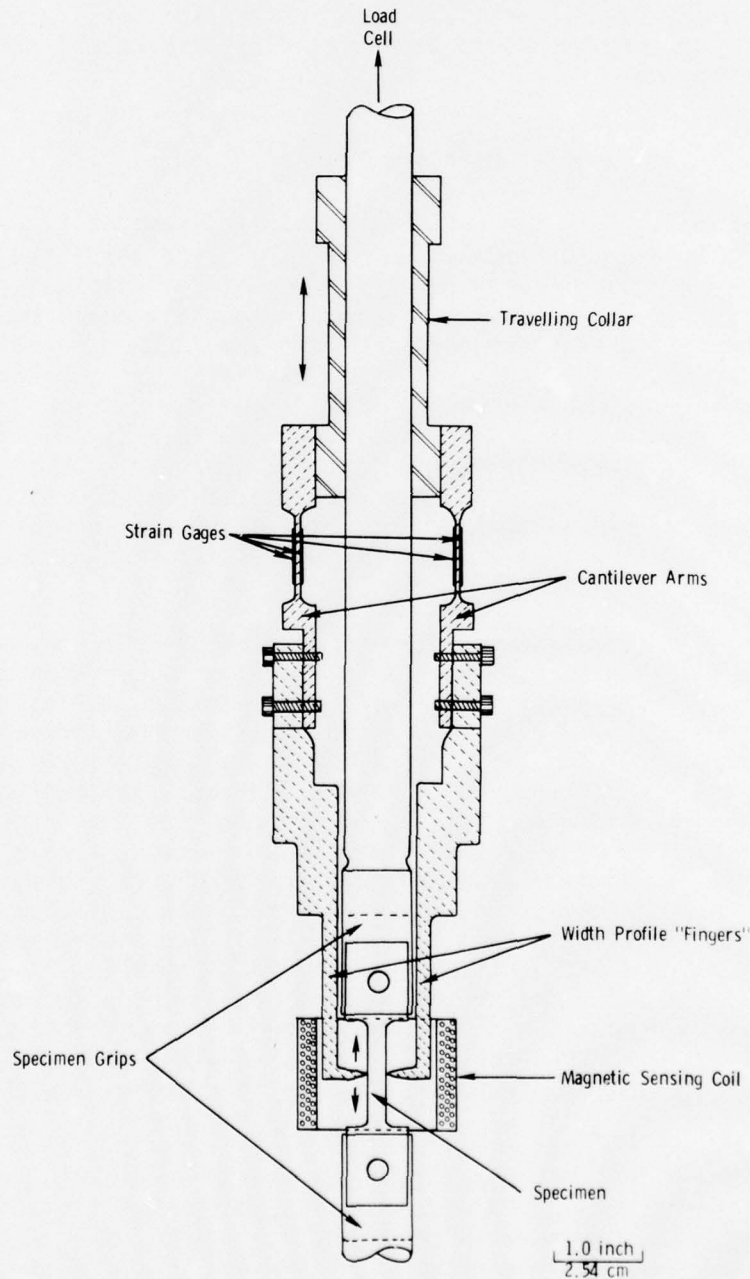


Figure 5. Geometry of experimental apparatus for simultaneous mechanical and magnetic measurements.

reinforced phenolic. To insure adequate response, the cantilever arm constructed of the same material. Because of the low thermal conductivity material, the strain gages were fan-cooled to insure stability. Only ferrite or paramagnetic materials were used near the sensing coil. Standard type 304 stainless steel clevis grips were used with titanium alloy grips. A load cell in series with the specimen and a linear potentiometer to measure crosshead displacement, four channels of a Sanborn strip-chart recorder simultaneously recorded the specimen load, crosshead displacement, specimen profile, and coil frequency.

### DATA ANALYSIS

During homogeneous deformation, the martensite content of a specimen was determined directly from the coil frequency drop after correcting for crosshead displacement effect due to the influence of the specimen grips. Since this effect was calibrated under zero load conditions, the crosshead displacement during a test was corrected for the machine compliance to accurately represent the displacement of the specimen grips. The true strain during homogeneous deformation was also obtained from the corrected crosshead displacement. To compensate for additional deformations outside the gauge length, these strain measurements were calibrated at the end of the test by measuring the cross section of the homogeneously deformed portion of the specimen and determining the true uniform strain. A small correction was sometimes made in the form of an "effective" gauge length varying linearly with crosshead displacement.

In the case of the inhomogeneous flow associated with Lüders-band formation, the specimen width profiles were converted to true-strain profiles as if a constant plastic anisotropy coefficient ( $R$ ). This  $R$  value was determined from the specimen width change in the fully developed Lüders band and the true strain obtained from the homogeneous strain measurement at the end of the Lüders band propagation. With many measurements of total martensite content (frequency drop) and the complete specimen strain profile, the function  $f(\bar{\epsilon})$  in principle can be determined precisely by iteration. However, within the experimental error limits, a measurement of *average* martensite content ( $\bar{f}$ ) versus average strain ( $\bar{\epsilon}$ ) in the forming Lüders band was considered sufficient for comparison. This would provide an accurate determination of  $f(\epsilon)$  for linear behavior. The error introduced being proportional to deviation from linearity. The function  $\bar{f}(\bar{\epsilon})$  would, however, depict the correct sense of any deviation from linearity, and this was the desired information to distinguish between linear and sigmoidal behavior in TRIP steels. It should be noted that the error introduced here is on an extremely local scale in the earliest stages of Lüders band formation.

The average strain ( $\bar{\epsilon}$ ) in a forming Lüders band was obtained from the strain profile. The frequency drop was taken to measure the total martensite content in the band. This represents the integral of the local martensite content throughout the band while the integral of the local strain is represented by the crosshead displacement ( $\Delta l$ ). Thus, the Lüders band volume necessary to convert total martensite content to  $\bar{f}$  was established by determining

band length ( $l_L$ ) necessary to account for  $\Delta l$  with a strain  $\bar{\epsilon}$  using the relation  $\bar{\epsilon} = \ln(1 + \Delta l/l_L)$ . Strains were sufficiently small so that the cross-sectional area can be taken as the original cross-sectional area  $A_0$ , giving an effective Lüders band volume of  $A_0 l_L$ . The measured frequency drop was then normalized to the volume of the total specimen gage section of length  $l_0$  by multiplying by the factor  $l_0/l_L$ , and was plotted with the values obtained during homogeneous deformation.

### STRAIN SENSITIVITY CORRECTION AND FINAL CALIBRATION

Corrected frequency drop versus strain curves during both Lüders-band formation and homogeneous deformation were obtained from tension tests on the TRIP steel in both the solution-treated and warm-rolled conditions. Tests were conducted at various temperatures from -196 C to 150 C by immersing the specimen assembly in suitable temperature baths. Tests below room temperature were carried out in alcohol or isopentane baths cooled with liquid nitrogen, or tested directly in liquid nitrogen. Above room temperature, hot silicone oil was used. The frequency versus strain curves for specimens in which a large amount of martensite formed at relatively high strains showed a maximum and "bent over" as if the martensite content were decreasing at high strains. The final martensite content of each specimen was determined by several techniques to be discussed. The specimens which showed the largest frequency reversal effect were found to contain 100% martensite after the test, indicating that this "bend over" must be due to a strain dependence of the coil sensitivity and not a reversion of martensite during the test. This effect is most likely due to a strain dependence of the martensite magnetic permeability. Studies of the influence of plastic deformation on the magnetic permeability of nickel have shown a linear dependence of permeability ( $\mu$ ) on plastic strain at high strains with a value of  $(1/\mu)(d\mu/d\epsilon)$  of approximately  $-1 \times 10^{-2}$  per 1% strain.<sup>14</sup> Similar behavior may be expected for TRIP steel martensite, particularly after the high strains associated with the prior thermomechanical treatment. Accordingly, a linear dependence of coil sensitivity on plastic strain was assumed.\* The magnitude of this strain dependence was evaluated by taking the final slope of the frequency curves of the completely martensitic specimens to represent the frequency change due only to the deformation of a fully martensitic material. The magnitude determined for both the solution-treated and warm-rolled materials was very similar to that reported for nickel.<sup>14</sup> The strain dependent correction factor thus defined was applied to all the frequency versus strain curves.

Due to the uncertainties introduced by the required corrections, the frequency measurements were used only to determine the shape of the transformation curves. Each test was individually calibrated by determining the amount of martensite in the uniformly deformed portion of the specimen gage section after the test. This was done by quantitative optical metallography, density measurements, and X-ray diffraction.

\*A further assumption is that plastic strain is the dominant factor affecting the martensite permeability, such that the influence of the changing elastic stress and crystallographic texture during the test can be neglected.

14. CULLITY, B. D. *Introduction to Magnetic Materials*. Addison-Wesley, Reading, MA, 1972, p. 152-155, 353.

Quantitative metallography was performed with a Quantimet optical analyzer. This technique is most accurate for coarse martensite plate structures since a lack of resolution can lead to an overestimate of martensite content for fine martensite structures as encountered in strain-induced transformations. More precise determinations of martensite content were made by measuring the density of the uniformly deformed portion of the gage section by the standard technique of weighing in air and in water. Densities could be determined within 0.2 percent. With a total transformation volume change of 4%, the martensite content could in principle be evaluated to within 5% martensite. Occasional surface cracks were a possible source of error in the density measurements, although a thorough cleaning and drying procedure was employed before weighings. Metallography and density determinations generally agreed within 20% and an average of the two measurements was adopted to calibrate the transformation curves obtained from the magnetic data. Due to a large grain size and preferred orientation, X-ray diffraction was employed only to verify the extremes of 100% martensite or austenite. Metallography alone was used to estimate the martensite content at fracture in specimens which did not exhibit any uniform elongation.

To test the accuracy of the transformation curves obtained after all the foregoing approximations and corrections, an independent determination was made of the room temperature transformation curve for material in the high-strength condition (80% prior reduction at 450 C) exhibiting Lüders-band behavior. Separate tension specimens were strained to different levels of Lüders-band development. The center portion of the developing band over which the strain was uniform within  $\Delta\epsilon = \pm 0.01$  was cut out with a low-speed diamond saw. The densities of these small pieces (approximately 0.2 x 0.1 x 0.1 inch or 0.5 x 0.25 x 0.25 cm) were obtained by precision machining all faces parallel within 0.0001 inch (0.00025 cm) to precisely determine the volume, and then weighing after thorough cleaning and drying. Figure 6 shows the room temperature  $f$  versus  $\epsilon$  curve resulting from the magnetic technique. The martensite content determined from the density measurements on individual specimens is represented as points for comparison. Within the expected error limits of the density measurements and possible variations between individual specimens, the agreement is quite good and it is concluded that the data derived from the magnetic technique do reasonably represent the true shape of the transformation curve.\*

It is evident that the transformation curve of Figure 6 has a positive (upward) curvature at low strains in contrast to the downward curvature required by the parabolic relation discussed earlier. The initial upward curvature is consistent with the sigmoidal behavior observed in low-strength metastable austenites. Having verified the accuracy of the room-temperature transformation curve with the individual density measurements, the magnetic technique represents a convenient method for determining the shape of the transformation curve with respect to true strain as a function of temperature during both Lüders-band formation and homogeneous deformation. The complete set of transformation curves and the associated true stress-strain curves will be presented in a separate report.

\*A possible source of error in the  $\alpha'$  martensite content determination by density measurement (particularly at low strains) would be the formation of hcp  $\epsilon'$  martensite which exhibits a transformation volume contraction on the order of 1%.<sup>5</sup> However, X-ray diffraction revealed no evidence of  $\epsilon'$  martensite in the specimens cut from the forming Lüders bands.

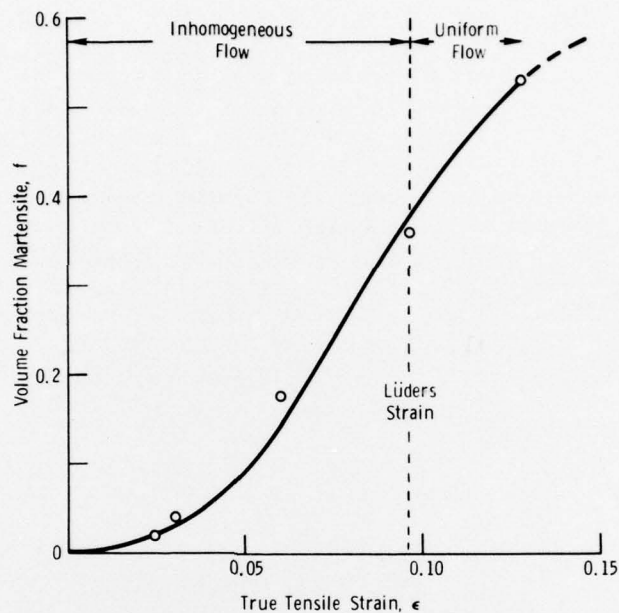


Figure 6. Transformation curve,  $f$  versus  $\epsilon$ , obtained at room temperature for high-strength TRIP steel. Solid curve is derived from continuous magnetic measurements during a single test. Open circles were obtained from density measurements from individual interrupted tests. Upward curvature during the initial period of inhomogeneous flow (Lüders-band formation) is clearly evident.

### CONCLUSIONS

A magnetic technique based on AC permeability has been combined with a method of local true-strain measurement to obtain volume fraction martensite versus true strain curves for TRIP steels during both Lüders-band formation and homogeneous deformation. Despite a number of required corrections and the need for extensive calibration, the technique is found to give an accurate determination of the transformation curve shape at room temperature, and represents a convenient method for studying the transformation behavior as a function of temperature. The transformation curve obtained for a high-strength TRIP steel during Lüders-band formation at room temperature is consistent with the sigmoidal type of behavior reported for low-strength metastable austenites.

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