Features of TRIP steel deformation at low and moderate temperatures

A V Frolova¹, V V Stolyarov², J V Tilak Kumar³ and J Sudha³

¹ Advanced Forming Research Centre, University of Strathclyde, 85 Inchinnan Drive, Renfrewshire, PA4 9LJ, UK
² Mechanical Engineering Research Institute of the Russian Academy of Sciences, 4, M. Kharitonyevskiy Lane, Moscow, 101990, Russia
³ Department of Mechanical Engineering, College of Engineering Guindy, Anna University, Chennai, 600025, India

anna.frolova@strath.ac.uk

Abstract. The TRIP effect and tensile behaviour of a TRIP steel were studied at temperatures ranging from -120 to 200°C. The initial microstructure comprises of austenite and martensite (50:50). Excellent combination of UTS (~2395 MPa) and elongation (28 %) was observed at -100°C and is attributed to the TRIP effect, that found absent above 100°C and below -120°C. Transformation temperatures were studied by DSC and DMA. Analysis of the tensile behaviour, phase changes and fracture surface suggest possible changes in the deformation mechanisms at different temperatures.

1. Introduction

The phenomenon through which martensitic transformation in metastable austenitic steels takes place during plastic deformation is called the TRIP effect (TRansformation-Induced Plasticity) [1]. Despite the limited understanding of its origins, scientific and practical interest in this phenomenon has remained high. The practical interest stems from the use of TRIP steels as structural materials in general engineering (high-strength, corrosion-resistant fasteners, wires, cables), aircraft parts (helicopter rotor torsions), instrumentation (springs and elastic elements of the thinnest sections), biomedical applications etc. [2], [3]. Such steels possess not only high corrosion resistance and weldability, but also a winning combination of high strength (about 2000 MPa) and ductility (up to 35%).

Scientific interest in the TRIP effect implies an understanding of the transformation kinetics and the features of the mechanism(s) causing the TRIP effect, as well as its control by internal (structure refinement, chemical and phase composition) and external (temperature, speed and degree of deformation during thermomechanical processing; cyclic loads, external fields) variables. Many of these factors have been studied quite thoroughly in previous works [4-11]. Some general ideas of developments in metastable austenitic steels are given in a review [4] and a book [5]. In addition to chemical composition, the important parameters controlling the phase transformation are grain size, degree and temperature of deformation [6-8]. It is demonstrated that the shear stresses and high pressures present during the refinement of the microstructure through intense plastic deformation by
torsion in AISI 304L steel can lead to the appearance of an intermediate phase ε (hcp), so that the sequence of transformations \( \gamma (\text{bcc}) \rightarrow \varepsilon \rightarrow \alpha' (\text{hcp}) \) is confirmed [6].

In places of deformation localization, hardening occurs due to the transformation of austenite (A) into martensite (M), and further deformation is concentrated in neighbouring volumes of austenite [7]. The completion of the phase transformation depends on the deformation temperature with respect to the critical temperatures, \( M_s \) (the start temperature of martensitic transformation upon cooling) and \( M_f \) (the maximum deformation temperature above which no transformation occurs) [7]. In accordance with previous studies [4], [7], [8], a distinctive feature of most TRIP steels is that \( M_s < 0^\circ \text{C} \), and \( M_f > RT \). Thus, even after quenching, the austenitic steel at ambient temperature remains as austenite. An increase in the degree of plastic deformation and refinement of the initial structure in AISI 304L metastable steel to the nano- and ultrafine- grain states contributes to an increase in the amount of martensite formed and additional hardening. However, ductility before fracture decreases [8]. In the metastable AISI 304 stainless steel, an increase in the tensile strain rate in the range of \( 10^{-4} \text{ s}^{-1} \) - \( 10^{4} \text{ s}^{-1} \) leads to a noticeable increase in Ultimate Tensile Strength (UTS) and yield strength (YS) [9]. In contrast, a decrease in the same properties with respect to an increase in the tensile strain rate approximately in the same range from \( 4.1 \cdot 10^{-5} \) to \( 2.5 \cdot 10^{-2} \text{ s}^{-1} \) in the austenitic – martensitic steel VNS9-Sh is also reported in literature [10]. With regard to external factors influencing the TRIP effect, it has been shown that the application of pulsed current during tensile deformation of the same steel led to a suppression of the TRIP effect [11].

Among the various factors that significantly affect the kinetics of martensitic transformation and, consequently, the TRIP effect, insufficient attention has been paid to the tensile test temperature [13-15]. Studies focusing on that, typically involve tests above ambient temperature. However, it is essential to include both, low and moderate, temperatures in order to determine the boundaries of the TRIP effect. An analysis of a few publications on similar studies in a wider temperature range, including very low and moderate temperatures, demonstrates a noticeable discrepancy in the results obtained [16-18]. For example, in [16] for TRIP Fe-Mn steels with addition of both, aluminium and silicon, the temperature dependence of increasing tensile elongation correlates with the energy of stacking faults and twinning, and the best plasticity is recorded at room temperature. However, in [17] for lightly alloyed TRIP steel 600 studied in the range from -60 to 120°C, it was shown that maximum plasticity is observed at 70°C. Note that in both those studies, the chemical composition of TRIP steels differs sharply from steels with high content of chromium and nickel. Finally, in a steel close in chemical composition to the Cr-Mn-Ni TRIP steel studied in this article, the authors found the maximum value of elongation at 100°C [18]. TRIP steel, developed at VIAM (Russia), with UTS up to 2000 MPa and ductility of up to 35%, is used in the production of high-strength fasteners, corrosion-resistant high-strength wires and cables, critical parts of aircraft (in particular, helicopter rotor torsions). The products from this steel display excellent characteristics of mechanical properties after cold plastic deformation, when the phase composition of the steel consists of approximately equal amounts of work-hardened austenite and strain-induced martensite (SIM). Therefore, TRIP steel is mainly used in the form of thin rolled sheets or wires.

The aim of this work is investigation and critical examination of the tensile deformation behaviour at low and moderate temperatures of a TRIP steel.

2. Material and methods
A 0.3 mm thick sheet made of VNS9-Sh steel was obtained by cold rolling of hot-rolled steel in several steps with intermediate heat treatment. The chemical composition is shown in table 1. The steel structure in the initial state was a mixture of SIM and work-hardened austenite (~ 50% martensite and ~ 50% austenite).

Quasistatic tensile tests of standard flat samples (gauge size 0.3x2.5x15 mm) were performed at temperatures in the range of -120 to 200°C on a horizontal tensile testing machine IR 5081-20 with a strain rate of \( \dot{\varepsilon} = 8.3 \cdot 10^{-4} \text{ s}^{-1} \). The desired temperature in the range of 20 - 200°C was achieved with an industrial dryer [15]. Tests at low temperatures were performed as follows: before the tensile test, a
chamber in the form of a tube with a hole cut out was mounted to the grips with the sample fixed between them (figure 1). Then the sample was cooled by immersing the assembly in liquid nitrogen for 3 minutes to stabilise the temperature. The required test temperature was achieved by varying the distance between the chamber and the sample surface that is exposed to the liquid nitrogen vapour in the range of 1 - 5 cm. Liquid nitrogen was topped up as and when required in an appropriate manner to compensate for losses. The temperature was monitored by a UT321 contact temperature meter with a chromel-alumel thermocouple attached to the centre of the gauge length and with an accuracy of ± 5°C.

![Figure 1. Test chamber with liquid nitrogen and a sample.](image)

The determination of phase transition temperatures was carried out by DSC using a calorimeter Sensys Evo (Setaram). The measurements were performed in the temperature range from -90 to 300°C at a speed of 3 deg·min⁻¹. The phase composition was determined by XRD analysis in CuKα radiation using a Rigaku Ultima IV X-ray diffractometer. XRD analysis was performed far from the place of final specimen fracture and the region between the grips section and the gauge part of the sample. The temperature dependence of internal friction was studied by Dynamic Mechanical Analysis (DMA) on a Q800 analyser using a three-point bending scheme in the temperature range from -150°C to 400°C with 1 mm amplitude and 1 Hz frequency. Driving Force was 3.8 - 3.1 N. The fracture surface of the tensile tested samples was studied by JSM-6610LV Electron microscope with accelerating voltage of 25 kV.

3. **Experimental results**

3.1. **X-Ray analysis**

Previously performed X-Ray analysis [19] clearly indicated that when the test temperature is reduced from 20 to -120°C, the relative intensity of the diffraction peak of austenite (220) drastically decreases and other two austenitic peaks (200) and (311) disappear. Simultaneously, the relative intensity of all the martensite diffraction peaks increases, including the strongest peak (211) (figure 2). The latter feature indicates the presence of martensite grains preferred orientation with crystallographic planes (110) parallel to the sample surface. Thus, when the test temperature drops below 20°C, the direct transformation of residual austenite to martensite increases, the volume fraction of which can reach 95% or more.

| Chemical element | C   | Cr  | Ni  | Mo  | Mn  | Si  | N   | S   | P   | Fe  |
|------------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Weight, %        | 0.25| 14.5| 4.8-5.8| 2.7-3.2| ≤ 1 | ≤ 0.6 | 0.03-0.07 | ≤0.01 | ≤0.015 | Bal. |

![Table 1. TRIP steel composition.](image)
3.2. Mechanical properties

Figure 3 shows the stress-strain curves at temperatures ranging from -120°C to 200°C. A yield stress and a strain hardening (SH) stage with a very low SH exponent can be observed at 23°C, which corresponds to a typical tensile curve when the TRIP effect is present [20]. Raising the test temperature to 100°C and above dramatically changes the shape of the deformation curves.

Figure 3. Stress-strain curves in the temperature range -120°C to 200°C.
There is no yield point, and the stage of SH has a reasonably high exponent. A significant decrease in the elongation at fracture ($\varepsilon$), as well as in the offset YS with an increase in temperature can be seen. The shape of the tensile curves at sub-zero temperatures indicates a significant difference in the SH exponent of the samples. In the range of 0 to -100°C, there are upper and lower yield points on the tensile curves, after which the stage of SH is observed. At a test temperature of -120°C, the upper and lower yield points are absent. Tensile properties at the different test temperatures are presented in table 2.

**Table 2. Mechanical properties at different temperatures.**

| Test temperature, °C | UTS, MPa | YS, MPa | $\varepsilon$, % |
|----------------------|----------|---------|-----------------|
| 200                  | 1440     | 800     | 2               |
| 100                  | 1505     | 810     | 7               |
| 50                   | 1610     | 900     | 16              |
| 23                   | 1830     | 1150    | 22              |
| 0                    | 1940     | 1200    | 23              |
| -20                  | 1925     | 1100    | 25              |
| -50                  | 2015     | 1170    | 29              |
| -70                  | 2220     | 1450    | 26              |
| -100                 | 2395     | 1560    | 28              |
| -120                 | 2460     | 1730    | 18              |

Figure 4 shows the dependence of the UTS, YS, $\varepsilon$, and the $\alpha$-phase content on the test temperature. A rise in the test temperature from ambient to 200°C leads to a rather sharp drop in UTS and ductility, as well as a decrease in the SIM formed (from 84% to 55%, respectively) (figure 4). A noticeable growth in UTS and YS and a slight increase in elongation were observed as temperature decreased. The maximum UTS (2460 MPa) was observed at -120°C (figure 4) with a sharp drop in ductility. The amount of SIM after deformation also grows with decreasing test temperature. At -100°C the volume fraction of martensite is 97% (comparing to initial ~50%). Maximum $\varepsilon$ (29%) was observed at -50°C, and then it continuously reduces with increasing temperature up to 200°C (figure 4).

![Figure 4. Temperature dependence of mechanical properties and $\alpha$-phase content.](image-url)
3.3. **DSC analysis in the initial state**

DSC analysis was used to determine the phase transformation temperatures in the initial dual-phase microstructure in the range from -90 to 300°C. During heating, two peaks (upward and downward) are seen in curve 1 (figure 5), following one after another: at 4.5°C (exothermic) and 11°C (endothermic). That could be associated with the start of the martensite decomposition into a ferrite-carbide mixture (upward peak) and the thermal transformation of residual austenite into martensite (downward peak). A repetition of the experiment with the same sample led to its reproducibility and a small shift of the peaks to a region of lower temperatures (figure 5, curve 2). No peaks were observed during cooling.

![Figure 5. The heat flow dependence on temperature during heating.](image)

3.4. **Fracture analysis**

Examination of the fracture surface (figure 6) showed a typical ductile dimple fracture across the test temperature range, however their size and presence of characteristic areas differ. The dimple size decreases with the test temperature, the dispersion becomes finer (figure 6 (a), (d)), the fracture surfaces appear rougher, and localised ductile shear regions are seen (figure 6 (d), (e)). At temperatures above 100°C, the fracture pattern begins to resemble the fracture surface of elevated temperature specimens.

4. **Discussion**

The reason for the slight deviation of the elastic modulus (figure 3), may be due to a difference in the rigidity of the “grips-sample” system, as well as the effect of nitrogen vapour or hot air on the material of the grips. Pronounced TRIP effect is observed at ambient temperature together with a low SH exponent and high ductility due to the phase transformation A → M. In samples tested at reduced temperatures, the SH exponent increases with deformation, as does the elongation at fracture. This may indicate a partial appearance of the TRIP effect at the early stages of deformation. The tensile curve at -120°C exhibits the maximum SH exponent as well as a slight decrease in ductility, which looks typical for a stable austenite phase. The shape of the stress-strain curves below -100°C indicates TRIP effect absence. Upper and lower yield points preceding the yield plateau can be seen in the range of 0 to -100°C. The high SH exponent at low temperatures can indicate a mechanism of additional SIM formation different from the necking mechanism at ambient temperature (compare curves at 23°C and 0°C in figure 3). In [20], the authors explained the presence of upper and lower yield points and the yield plateau in metastable austenitic steels by three features of the plastic deformation
mechanism, viz. slip, twinning and martensitic transformation. In [21], it was noted, that TRIP steel VNS9-Sh does not exhibit the physical YS when it is in purely austenitic and purely martensitic states. That is consistent with disappearance of the upper and lower yield points in present study when the test temperature is decreased to -120°C (figure 3), as well as the increasing amount of α-phase (martensite) with temperature reduction (~ 95% at -120°C) (figure 4). The absence of TRIP effect features in the tensile curves at low temperatures (yield points, plateau and a low degree of SH) indicates the TRIP effect disappearance.

Figure 6. The sample fracture surfaces after tensile tests at different temperatures: red arrows indicate micro-cracks; white circles indicate ductile shear regions.

At a test temperature of 50°C, the amount of SIM is more than in the non-deformed initial material, which indicates that at this temperature the TRIP effect is present (formation of additional SIM). In the temperature range of 100 – 200°C, the deformation mechanism changes due to a partial reverse transformation of martensite to austenite, which is consistent with previous studies [15]. According to [22], the complete reverse M→A transformation in VNS-9Sh steel occurs after a tempering treatment at 600°C.
DSC analysis made it possible to experimentally establish that the temperature of the thermal stability of SIM is noticeably below the ambient temperature. Considering the possible temperature hysteresis, it can be assumed that the Mₜ temperature of the direct austenite to martensite transformation during cooling is close to 0°C or lower, which is consistent with published data [4], [7], [8]. Figure 4 confirms this assumption, since the increase in the volume fraction of martensite is practically exhausted for T ≤ 0°C. In addition, especially for temperatures equal to and below 0°C, the degree of SH sharply increases (figure 3).

The microstructure of the fracture surfaces demonstrates the ductile nature of fracture, mainly by dimple fracture, which ensures good ductility, despite an increase in the martensite content. However, the size of dimples decreased with a lowering of test temperature and may be responsible for the increase in the tensile strength. Another feature of fracture is the appearance of ductile shear regions (figure 6 (d), (e)) and formation of micro-cracks (figure 6 (c), (f)).

5. Conclusions
The features present in the stress-strain curves (yield points, SH) and the mechanical properties of metastable austenitic TRIP steel in the temperature range of -120 to 200°C are reported.

The tensile behaviour at low and moderate temperatures, a change in the austenite/martensite phase ratio, and fracture analysis indicate drastic changes in the operating deformation mechanisms at different temperatures and, accordingly, the contribution to the TRIP effect.

An increase in the test temperature above the ambient leads to a decay in SIM formation. A decrease in temperature assists the formation of additional SIM. Changes in the phase composition lead to a decrease in the TRIP effect contribution at elevated (> 50°C) and reduced (<0°C) test temperatures and its disappearance at temperatures above 100°C and below -120°C.

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