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Combining gradient structure and TRIP effect to produce austenite stainless steel with high strength and ductility

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ABSTRACT

We report a design strategy to combine the benefits from both gradient structure and transformationinduced plasticity (TRIP). The resultant TRIP-gradient steel takes advantage of both mechanisms, allowing strain hardening to last to a larger plastic strain. 304 stainless steel sheets were treated by surface mechanical attrition to synthesize gradient structure with a central coarse-grained layer sandwiched between two grain-size gradient layers. The gradient layer is composed of submicron-sized parallelepiped austenite domains separated by intersecting ε-martensite plates, with increasing domain size along the depth. Significant microhardness heterogeneity exists not only macroscopically between the soft coarse-grained core and the hard gradient layers, but also microscopically between the austenite domain and ε -martensite walls. During tensile testing, the gradient structure causes strain partitioning, which evolves with applied strain, and lasts to large strains. The $\gamma \to \alpha'$ martensitic transformation is triggered successively with an increase of the applied strain and flow stress. Importantly, the gradient structure prolongs the TRIP effect to large plastic strains. As a result, the gradient structure in the 304 stainless steel provides a new route towards a good combination of high strength and ductility, via the co-operation of both the dynamic strain partitioning and TRIP effect.

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1. Introduction

It has been a challenge to produce metals and alloys with both high strength and high ductility $[1-5]$ $[1-5]$ $[1-5]$. High strength can be achieved by well-known strategies such as grain refinement, deformation twinning, nano-twins, second-phase particle strengthening, solution hardening, and work hardening. However, this usually comes with sacrifice of ductility $[4-9]$ $[4-9]$. Ductility is measured under tensile loading either as total elongation to failure or as uniform elongation. The decrease in ductility with increasing strength is often observed because i) high strength metals often have low strain hardening rate, as in the case of cold worked metals and nanostructured metals $[1,3-7]$ $[1,3-7]$ $[1,3-7]$; ii) according to the Considere criterion, a high-strength metal would need a higher strain hardening rate to reach the same uniform elongation as that of a lowstrength metal [\[8,10\].](#page--1-0)

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A promising strategy for simultaneously improving the strength and ductility is proposed recently by combining several plastic deformation and strain hardening mechanisms in steels $[11-15]$ $[11-15]$. Each hardening mechanism may be active over a certain strain range, but several of them together could collectively produce a high strain-hardening rate to improve ductility, when at least one of them persists over a wide range of the imposed tensile strains [\[11,12\].](#page--1-0) One of the most effective approaches to increase both strain hardening and ductility is the transformation-induced plasticity, i.e. the TRIP effect, which is observed in dual-/multi-phase steels $[11–13,16–18]$ $[11–13,16–18]$ $[11–13,16–18]$ $[11–13,16–18]$ $[11–13,16–18]$, TRIP steels $[11,12,19–23]$ $[11,12,19–23]$, bainite steels $[15,24]$, etc. In these steels, the phase-specific deformation mechanism takes effect at different stress or strain levels and therefore, the global plastic response is composite-like, which critically depends on the evolution of stress transfer and dynamic strain partitioning among different phases [\[11,12\].](#page--1-0) In addition, martensitic transformation is activated at varying local stress or strain levels during the tensile tests, which provides not only higher strain hardening but also excellent plasticity, when the TRIP effect is designed to sustain over Exection plasticity, when the TRIP effect is designed to sustain over
E-mail address: xlwu@imechac.cn (X L Wu) a wide strain range [\[11,12\]](#page--1-0). In addition, to produce good TRIP effect,

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the phase transformation needs to persist up to large strains. High strain hardening rate is reported to originate from the harder phase produced by phase transformation as well as mechanical incompatibility, i.e. strain partitioning, between different phases with different flow stresses $[11-13,16]$ $[11-13,16]$ $[11-13,16]$. Strain partitioning causes strain gradient at phase boundaries, which is accommodated by geometrically necessary dislocations [\[25,26\]](#page--1-0) and consequently enhances strain hardening [\[27\].](#page--1-0) Recently, a microstructural design concept, referred to the TRIP-maraging steel, is proposed [\[11\].](#page--1-0) The strength and ductility were improved simultaneously from three effects, i.e. the TRIP effect by nano-scale austenite, maraging effect by nano-precipitation, and composite effect due to strain partitioning.

Recently, another approach employing gradient structure (GS) has been reported to produce a superior combination of yield strength and ductility $[28-34]$ $[28-34]$. The GS often consists of the grainsize gradient layers (GLs) with increasing grain sizes along the depth towards the central coarse-grained (CG) core layer. For gradient structured metals with stable gradient structures, high ductility is attributed to an extra strain hardening due to the presence of strain gradient and the change of stress states, which generates geometrically necessary dislocations and promotes the generation and interaction of forest dislocations [\[29\].](#page--1-0) The GS may produce an intrinsic synergetic strengthening, with its yield strength much higher than the sum of separate gradient layers [\[34\].](#page--1-0) This is due to the macroscopic strength gradient and mechanical incompatibility between layers $[29-34]$ $[29-34]$ $[29-34]$. Such a mechanical incompatibility was also found to produce high back stress, which may contribute to both strength and ductility [\[3,35\].](#page--1-0)

The objective of this investigation is to propose a design strategy, referred to as the TRIP-gradient steel, to produce high strength and high ductility. The 304 austenite stainless steel (304ss) is chosen as the model material due to its TRIP effect during tensile testing $[36-44]$ $[36-44]$. The mechanical properties of 304ss have been extensively studied $[45-48]$ $[45-48]$ $[45-48]$. In early studies, gradient structure in 304ss was reported to produce a high yield strength of 950 MPa with a ductility of 30% [\[49,50\]](#page--1-0), but its plastic deformation and strain hardening mechanisms were not explored. In this work, we combined the gradient structure with the TRIP effect in 304ss. In particular, the TRIP effect was designed to operate in a wider strain range, which depends on dynamic strain partitioning associated with the composite-like deformation in the GS. Microstructural observations and measurements were performed on tensile deformed samples to correlate the excellent strength and ductility to the strain partitioning and TRIP effect.

2. Experimental procedure

2.1. Materials and SMAT process

In the present investigation, as-annealed AISI 304ss disks were used, with a diameter of 100 mm and thickness of 0.5, 1 and 2 mm, respectively. The chemical composition was 0.04 C, 0.49 Si, 1.65 Mn, 7.8 Ni, 16.8 Cr, 0.37 Mo and the balance Fe (all in mass%).

The surface mechanical attrition treatment (SMAT) technique was used to produce the gradient structured 304ss specimens, with a central CG layer sandwiched between two gradient layers (GLs). During the SMAT process, spherical steel shots of 3-mm in diameter were accelerated to high speeds using high-power ultra-sound to impact on the sample disk [\[51,52\].](#page--1-0) Because of the high frequency of the system (20 kHz), the entire surface of the component is peened with high density of impacts over a short period of time. The SMAT processing time was the same for both sides of each disk, which

was $1-15$ min. No crack was observed on the sample surface after SMAT processing.

2.2. Mechanical property tests

Tensile specimens were cut from the SMAT-processed disks. They are dog-bone shaped with a gauge length of 15 mm and width of 3 mm. Specimens of varying thicknesses in the gradient layers were also prepared for both tensile testing and X-ray diffraction (XRD) measuements of phase transitions at different depths and applied tensile strains. These specimens were prepared by polishing away the whole SMAT-processed sample from one side via mechanical polishing at first and later electro-polishing of at least $150 \mu m$ thick at room temperature, leaving behind the specimen of desired thickness.

Uniaxial tensile tests were carried out at a strain rate of 5×10^{-4} s⁻¹ at room temperature using an MTS Landmark testing machine. An extensometer was used to measure the strain during tensile testing. Uniaxial tensile stress-relaxation tests were also performed under strain-control mode at room temperature. Upon reaching a designated relaxation strain, the strain was maintained constant while the stress was recorded as a function of time. After the first relaxation over an interval of 60 s, the specimen was reloaded by a strain increment of 0.6% at a strain rate of 5×10^{-4} s⁻¹ for next relaxation. Three stress relaxations were conducted for each designated strain. All tensile tests were performed three times on average for each condition to verify the reproducibility of the monotonic and cyclic tensile stress-strain curves.

Vickers microhardness (Hv) indentations were made in the gauge section to measure the Hv evolution, prior to and after tensile testing respectively, under a 25 g load across the cross-section of the gradient structured specimen.

2.3. Microstructural characterization

Transmission electron microscopy (TEM) observations were conducted to investigate microstructural evolution in the GS samples during tensile tests in a JEM 2010 microscope with an operating voltage of 200 kV. TEM disks were cut from the gauge section of tensile samples after suspension of tensile testing at designated strains.

Electron back-scattered diffraction (EBSD) observations were conducted using a high-resolution field emission Cambridge S-360 SEM equipped with a fully automatic Oxford Instruments Aztec 2.0 EBSD system (Channel 5 Software). A scanning step of $0.03-0.08$ µm was performed during the EBSD acquisition. Due to spatial resolution of the EBSD system, the collected Kikuchi patterns can be obtained automatically at a step resolution of $0.02 \mu m$ and correspondingly misorientations less than 2° cannot be identified. The samples for EBSD examinations were mechanically polished carefully followed by electro-polishing using an electrolyte of 90 vol% acetic acid and 10 vol% perchloric acid with a voltage of 40–45 V at about -40 °C in a Struers LectroPol-5 facility.

X-ray diffraction (XRD) measuements were performed to obtain the phase transfor- mation information during tensile tests using a Philips Xpert X-ray diffractometer with Cu K α radiation. The samples were carefully prepared by mechanical polishing plus electropolishing from the surface to reach the designated depths after suspending tensile testing at varying designated strains. The phase volume fraction was estimated from the peak integrated intensities I_{hkl} after background subtraction. The volume fraction of α' -
martensite a Marko Calculated using the equation martensite, V_{α} , was calculated using the equation, $V_{\alpha'} = I_{bcc(110)}/I_{bcc(110)} + I_{fcc(111)}$

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