

EFFECT OF PLASTIC DEFORMATION INDUCED BY PRESSURE AND DRECE TECHNIQUE ON THE STRUCTURE AND MAGNETIC PROPERTIES OF AUSTENITIC STEELS

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Abstract

The study is focused on changes in the structural and physical properties of AISI 304 austenitic stainless steel subjected to severe plastic deformation at room temperature. The steel was deformed using two methods: applying compressive forces of 100 kN and 150 kN and using the Dual Rolling Equal Channel Extrusion (DRECE) technique. The presence of δ -ferrite and/or the partial transformation of paramagnetic austenite into ferromagnetic martensite were evidenced by X-ray diffraction, which confirmed 6 wt.% and 14 wt.% ferromagnetic components after the application of a 150 kN compressive force and DRECE at a surface depth of about 10 μm . The structural changes were also reflected in the bulk magnetic properties of the steel. Compared to the as-cast state, magnetization in a magnetic field of 2 Tesla was approximately six and three times higher after the application of a 150 kN force and DRECE, respectively. These results were supported by metallographic analysis. Recovery of the fully paramagnetic state was achieved after annealing the steel at 950 °C for 30 minutes. The comprehensive experimental characterization of the steel provides valuable information for its usage in practical applications.

Keywords: Austenitic Steel, Microstructure, Magnetism, Martensite, Pressure, DRECE

1. INTRODUCTION

The complex physical properties of austenitic steels, such as resistance to corrosion and high-temperature oxidation, energy absorption, and the ability to maintain mechanical properties over a wide temperature range, are currently being applied in many areas, including petrochemicals, gas turbines, aerospace, automotive, and healthcare [1-4]. Thanks to this combination of physical properties and their paramagnetic behavior, stainless steels can be used in battery components of currently developed electric vehicles [5].

Austenitic stainless steels typically contain at least 8 wt.% of nickel and 17 to 20 wt.% of chromium. Their paramagnetic nature is characterized by a low magnetic permeability of about 1.01-1.02. An increase in the permeability may be related to the presence of ferritic or martensitic phase in the structure and depends on the chemical composition of the steel and the conditions of heat and plastic processing. Two cases of austenite

to martensite transition can occur. Austenite can undergo a martensitic transformation because of a drop in temperature below the martensitic transformation starting temperature, depending on the chemical composition, or due to cold deformation, such as shaping of finished products, which involves strong mechanical strengthening (e.g. during bending, machining, or forming by drawing) [6]. The martensitic phase is ferromagnetic, resulting in increased magnetic effects of austenitic steels, which limit their ultimate applicability. Generally, steels with a low concentration of austenitic elements (nickel, manganese, carbon, and nitrogen) are more susceptible to martensitic transformation. Conversely, steels with increased nickel content show only slight changes in magnetic properties during cold forming [6].

This work focuses on commercial austenitic stainless steel AISI 304. Due to its good weldability, cold ductility, and resistance to temperatures up to 300 °C, this steel is appropriate for fuel tanks, exhausts systems, chassis for buses and trucks, and more [7,8]. The aim is to expand the incomplete information in steel material sheets regarding structural and magnetic properties. Therefore, the steel is subjected to cold deformation using pressure and Dual Rolling Equal Channel Extrusion (DRECE) technique and a possible paramagnetic-ferromagnetic transformation is studied by wide spectrum of experimental methods (metallography, X-ray diffraction, and magnetometry).

2. EXPERIMENTAL

The paper demonstrates the influence of plastic deformation induced by pressure and Dual Rolling Equal Channel Extrusion technique on the structural and physical properties of AISI 304 steel. The steel was produced by the Argon Oxygen Decarburization (AOD) process and its chemical composition was determined by glow discharge optical emission spectrometry (GDOES, wt.%): Cr – 17.91, Ni – 8.04, Mn – 1.77, Mo – 0.23, Si – 0.33, Cu – 0.29, Co – 0.15, W – 0.18, Nb – 0.03, C – 0.022.

For the pressure-induced deformation the steel was supplied in the form of a rod with a length of 3000 mm and a circular cross-section of 12 mm. Subsequently, the rod was turned into cylinders with a length of 15 mm. Solution annealing was carried out at a temperature of 1030 °C for 0.5 h in a vacuum atmosphere ($\approx$ 0.015 mbar) followed by cooling with gaseous nitrogen to room temperature (time of cooling $\approx$ 10 minutes). The annealed steel samples in the as-cast (AC) state were compressed with forces of 100 kN and 150 kN according to **Figure 1a**, resulting in a change in their dimensions. Deformations of 100 and 150 kN caused a reduction in the height of the cylinder to 12 mm and 10.7 mm and an increase in its diameter to 12.9 mm and 14.2 mm.

For the DRECE deformation the steel was supplied in the form of a cold-rolled sheet strip with a length of 1000 mm, a width of 58 mm and a thickness of 2 mm. In this case the steel was not annealed, so the as-cast state for DRECE differs from the as-cast state used for compression deformation described in the previous paragraph. The produced strip was processed by the DRECE technique schematically shown in **Figure 1b**. Details about this method can be found, for example, in Ref. [9]. Due to significant steel strengthening only one pass of DRECE was applied.

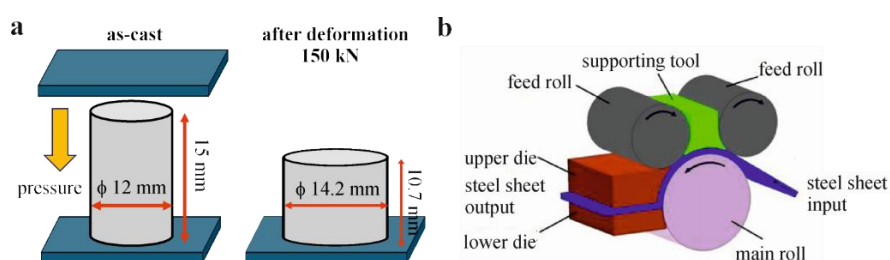


Figure 1 Forming of the steel samples by pressure (a) and DRECE technique (b)

A total of five steel samples were studied: AC states before compression deformation and DRECE, and deformation states after compression of 100 kN and 150 kN, and after DRECE. All samples were cut and machined according to the demands of individual experimental methods. The cutting was done using a wire cutter to exclude the undesirable structural and chemical changes due to a local thermal heating.

Metallographic analysis of the structure was carried out using an Olympus IX70 microscope. Observations were done on the steel longitudinal cross-section relative to the pressure and DRECE directions. The crystal structure was studied at room temperature (RT) using X-ray diffraction (XRD). We used AXS D8 Advance (Bruker AXS GmbH) diffractometer equipped with CuK α radiation ($\lambda = 0.1540598$ nm) and a position-sensitive LynxEye detector. The diffractograms were measured in 2θ range 35° - 95° with a step 0.014° and a time per step 2 s. The Rietveld structure refinement method and the ICDD PDF-2 database were applied to analyse the measured XRD spectra. The bulk magnetization curves of samples were measured at RT using a vibrating sample magnetometer (VSM, MicroSense, EZ9) in an applied magnetic field of ± 1600 kA/m.

3. RESULTS AND DISCUSSION

The different microstructure between the samples before and after pressure deformation is shown in **Figure 2**. **Figure 2a** visualizes the cross section of the AC sample after solution annealing. The microstructure consists of austenitic grains of various sizes with a polyhedral shape. Both annealing twins and non-ferrous inclusions based on sulphide, specifically manganese sulphide (MnS), are present. These MnS inclusions are arranged in rows along the rolling direction.

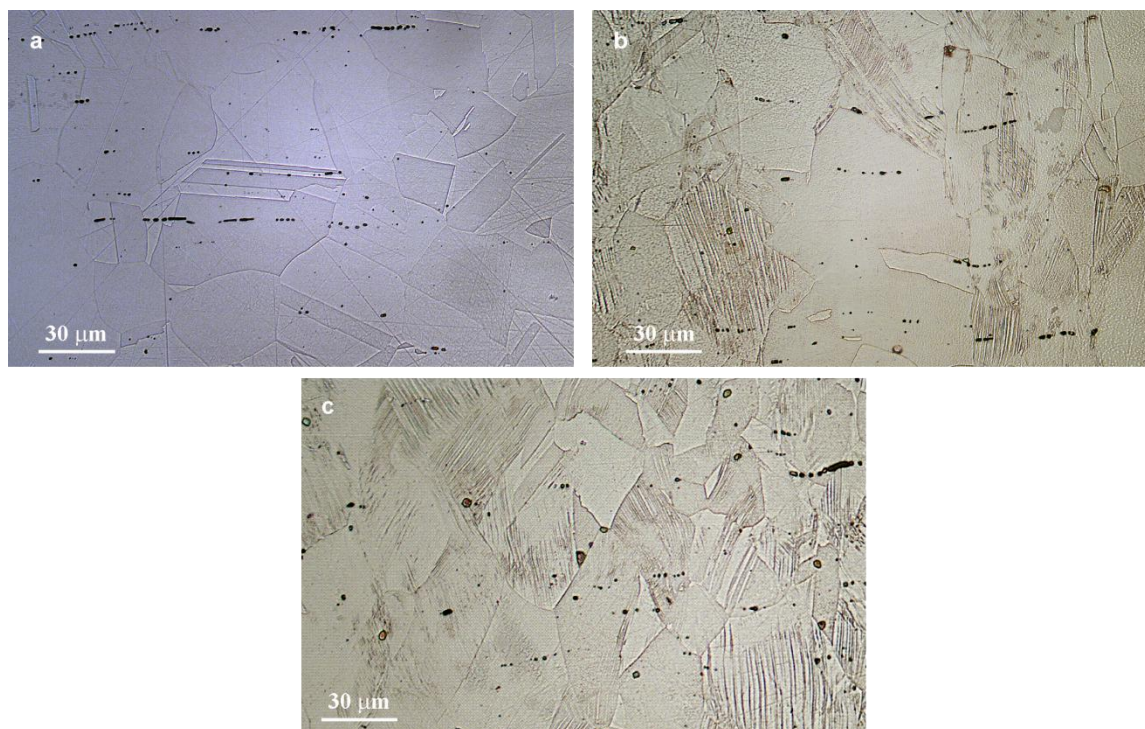


Figure 2 Scanning micrographs of the cross sections prepared in the longitudinal direction with respect to pressure for the AC (a), 100 kN pressed (b), and 150 kN pressed (c) steels

However, the microstructure after 100 kN pressure deformation, shown in **Figure 2b**, differs significantly. The polyhedral austenitic grains of various sizes and annealing twins can still be distinguished. MnS inclusions are arranged in rows, but their arrangement starts to appear more random. During plastic deformation, slip occurs in the austenitic grains, which is manifested by slip bands in individual the most favourably oriented grains.

Deformation martensite may be present in these areas. On the other hand, delta ferrite was not observed in the structure. A similar structure was observed in the sample after 150 kN pressure deformation (**Figure 2c**).

The microstructure of the steel samples before and after DRECE is comparable to those in **Figures 2b** and **2c**. The main grain size is about 30 μm , and deformation bands in the bulk and on the surface are more pronounced after the DRECE. Contrary to the pressure deformation, the narrow lines of delta ferrite are observed in the bulk. More information about microstructure of samples after DRECE forming can be found in reference [10].

Table 1 The results of Rietveld analysis of investigated steels in as-cast (AC) states and after deformation.
a – lattice parameter, *I* – phase content.

steel	state	<i>a</i> (nm)		<i>I</i> (wt.%)	
		austenite	δ -ferrite	austenite	δ -ferrite
AISI 304	AC pressure	0.3600(1)	0.2878(8)	95.3	4.7
	AC DRECE	0.3592(9)	0.2878(1)	89.7	10.3
	pressure 100 kN	0.3598(9)	0.2875(4)	95.4	4.6
	pressure 150 kN	0.3598(5)	0.2877(6)	93.9	6.1
	DRECE	0.3594(4)	0.2878(2)	85.6	14.4

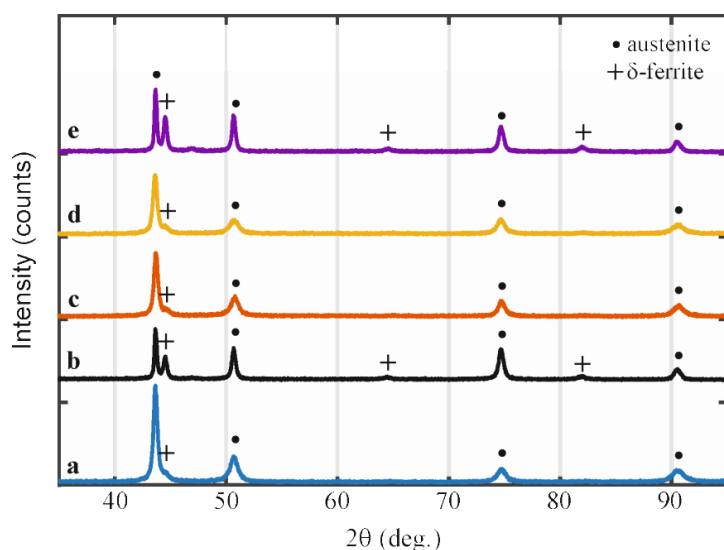


Figure 3 Room temperature XRD patterns of steel samples: as-cast states before pressure (a) and DRECE (b), after deformation by compressive forces of 100 kN (c) and 150 kN (d), and by DRECE (e)

The results of the metallography are supplemented by the XRD measurements presented in **Figure 3** and **Table 1**. The analysis of the measured diffractograms from the surface layers, approximately 10 μm thick, confirms a dominant contribution of the austenite (paramagnetic) phase in all studied samples. The ferromagnetic contribution is labeled as δ -ferrite in **Fig. 3** and **Table 1**, but it could be martensite, because δ -ferrite and martensite cannot be distinguished from the diffractogram analyses. As can be seen the samples in the as-cast state significantly differ in the amount of ferromagnetic phase. The AC sample for pressure deformation was annealed, which reduced the amount of ferromagnetic contribution compared to the non-annealed AC sample for DRECE, where this contribution likely increased due to cold rolling. After deformation, there was a significant increase in the amount of δ -ferrite after one pass of DRECE, specifically from 10.3 wt.% (AC state) to 14.4 wt.%. In contrast, the compressive force had a smaller effect on the amount of ferromagnetic phase, with no increase from 4.7 wt.% (AC state) to 4.6 wt.% at a pressure of 100 kN and higher increase to

6.1 wt.% at a pressure of 150 kN. It is also important to note that the uncertainty in phase determination is relatively high and can be up to $\pm 1\%$.

The bulk magnetization curves were measured by applying the external magnetic field ± 1600 kA/m and are shown in **Figure 4**. Smaller samples with a diameter of 5 mm and a thickness of 1 mm were cut from cylindrical steels before and after pressure deformation. The magnetic field was generated along the 5 mm base, perpendicular to the applied pressure. The magnetization curve of the as-cast sample in **Figure 4a** is almost linear, indicating a dominant paramagnetic contribution due to solution annealing. However, the non-zero hysteresis with a coercive field of about 14 kA/m points to a weak ferromagnetic component. After deformation by a compressive force of a 100 kN, the magnetization curve consists of a linear paramagnetic contribution at higher magnetic fields and a ferromagnetic contribution with magnetization reversal at lower magnetic fields (**Figure 4b**). The magnetization values in zero and maximum magnetic fields ($M_r \approx 0.21$ Am²/kg and $M_s \approx 1.42$ Am²/kg) significantly increased compared to the as-cast state, while the coercive field (H_c) slightly decreased to 12.4 kA/m. Similar trend continues even after the application of a compressive force of 150 kN (**Figure 4b**), when the magnetization values M_r and M_s are approximately three times higher compared to the force of 100 kN and H_c decreased to 9.8 kA/m. These results indicate an increase in the martensitic (ferromagnetic) phase with increasing compressive force. From the magnetic point of view, the martensite becomes magnetically softer with increasing deformation pressure.

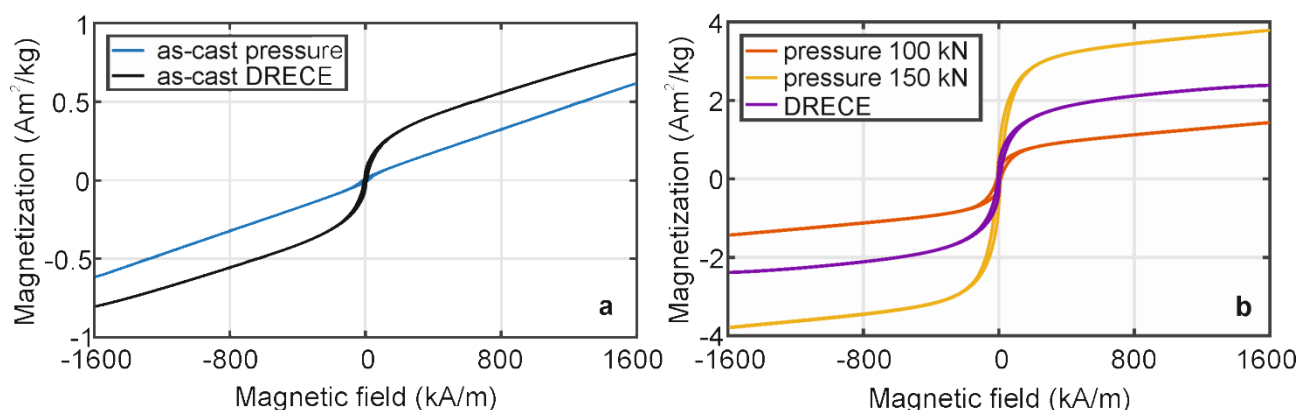


Figure 4 Magnetization curves of steel samples (a) in as-cast states and (b) after deformation by compressive forces of 100 kN and 150 kN and by DRECE.

From steel sheets before and after the use of DRECE, $(5 \times 5 \times 2)$ mm³ samples were cut, and their magnetization curves are shown in **Figure 4**. The magnetic field was applied parallel and perpendicular to DRECE direction, but the results were practically identical. The sample in as-cast state (**Figure 4a**) already contains a more pronounced ferromagnetic component, in good agreement with XRD results. After one pass of DRECE, the measured magnetization curve lies between the curves for compressive forces of 100 kN and 150 kN (**Figure 4b**), thus the amount of ferromagnetic martensite can be considered comparable. The obtained magnetic parameters are $M_r \approx 0.38$ Am²/kg, $M_s \approx 2.38$ Am²/kg, and $H_c \approx 6.34$ kA/m.

4. CONCLUSION

The present studies have aimed at the stability of the austenitic structure and the paramagnetic behaviour of the stainless steel AISI 304. It has been shown that deformation by pressure and using the DRECE method leads to the formation of deformation-induced martensite. The obtained experimental results can be summarized as follows:

- 1) **Pressure deformation:** The sample in original (AC) state was annealed at a temperature of 1030 °C for 0.5 hours before pressure deformation. Despite this, surface-sensitive and bulk-sensitive methods revealed a weak ferromagnetic contribution. The ferromagnetic response was significantly enhanced after deformation by a compressive force of 150 kN. The sample compressed by a smaller force of 100 kN exhibited a similar amount of martensite at a depth of approximately 20 µm below the surface as the AC sample, and only metallography on the cross-section and bulk magnetic measurements demonstrated its higher content in the volume. The change in martensite content depending on higher deformation forces will be the subject of further investigation.
- 2) **DRECE forming:** The sample in the AC state was not annealed and exhibited a more pronounced ferromagnetic contribution both on the surface and in the volume, which increased after DRECE deformation. Martensite and δ-ferrite were detected in both samples. The magnetization curve with comparable magnetic response suggests that one pass of DRECE in AISI 304 steel could correspond to a pressure of 100 to 150 kN.
- 3) Complete restoration of paramagnetic properties after both methods of deformation was achieved by annealing at a temperature of 950 °C for 0.5 hours.

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