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Effect of high temperature annealing on texture and microstructure on an AISI-444 ferritic stainless steel

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Abstract

AISI 444 is a Mo-alloyed ferritic stainless steel which presents good naphthenic corrosion resistance, making it attractive for applications in petroleum refining plants; however, good formability is also important. To achieve good formability with this alloy the annealing process is crucial. The annealing temperature in ferritic stainless steel is usually around 850 °C, which falls in the range of sigma phase precipitation. A means to avoid this precipitation is to anneal at temperatures around 1000 °C followed by rapid cooling. Annealing at high temperatures can cause grain growth and carbide or nitride precipitation which can result in a reduction of room temperature toughness. In this paper, the rolling and recrystallization textures of AISI 444 steel were studied in samples cold rolled with different thickness reductions (30%, 60%, 80% and 90%) followed by annealing at 955, 980 and 1010 °C. Aspects of grain size and carbide precipitation after annealing were characterized using EBSD and AFM. The material drawability was analyzed through strain rate or Lankford (*r*) coefficients calculated from texture results.

Keywords: Ferritic stainless steel; Rolling texture; Recrystallization texture; Carbide precipitates

1. Introduction

The evolution of cold rolling texture and recrystallization texture of ferritic stainless steels with 17% Cr has been studied by a number of authors [1–3]. The recrystallization temperature in most of these studies was in the range of 850 to 950 °C. The annealing temperature cannot be increased too much because the higher the annealing temperature, the lower the room temperature toughness [4].

The general corrosion resistance of AISI 444 is comparable to that of austenitic AISI 316. It is also very resistant to stress corrosion cracking and the very low content of nickel results in good properties in sulphur-containing environments at high temperatures [5]. These properties recommend the use of this steel in petroleum refining plants, replacing austenitic grades 316 and 317 L. For these applications good formability is important. The most commonly used method in the high volume production of complex three-dimensional work pieces is deep-drawing. Deep-drawing ability can be estimated by the Lankford value (*r*), which is defined as the ratio of the true strain in the width direction to the true strain in the thickness direction. A mean *r* value, r_m, is defined by measuring Lankford values (*r*) in the

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Table 1
Chemical composition of AISI 444 (wt.%)

% C	% Cr	% Ni	% Mo	% Ti	% Si	% N	% Nb	% Fe
0.015	17.56	0.20	1.86	0.13	0.54	0.0123	0.20	Balance

rolling, transverse and diagonal directions and is calculated as in Eq. (1) [6]:

$$r_m = 1/4(r_{0°} + 2r_{45°} + r_{90°}) \quad (1)$$

The variation of r value around the rolling plane is called Δr and is defined by:

$$\Delta r = 1/2(r_{0°} - 2r_{45°} + r_{90°}) \quad (2)$$

Typically, r_m values are around 1.0 for austenitic stainless steels. Low carbon steels and interstitial-free Ti-alloyed carbon steels can present r_m values around 2.0, which is preferable for drawing operations.

Deep drawing operations demand a sheet material that is highly compatible with deformation, so annealed material is normally used.

Raabe and Lücke [2] studied the texture of ferritic stainless steels containing between 11% and 17% Cr in four different through-thickness layers reflecting inhomogeneity of texture and microstructure through the sheet thickness. They concluded that cold rolling leads to a strong α fibre texture with a maximum at texture in the central layer. These authors also studied the effect of annealing at 850 and 950 °C in samples cold rolled 50% and 70% in thickness and found that the recrystallization texture of cold rolled samples with strong {111} <112> textures leads to a Goss texture after recrystallization. The required texture for deep drawing steels is a {111} fibre texture with parallel normal direction [7].

In the present work the evolution of texture in the center layer of AISI 444 samples after deformation and annealing over the temperature range from 950 to 1010 °C was analyzed. The results were related to the variation of the r value (Lankford parameter). Microstructural features after annealing were also investigated by electron back scattering diffraction (EBSD) and atomic force microscopy (AFM).

2. Experimental

The chemical composition of the AISI 444 steel studied is shown in Table 1. The steel was industrially hot rolled to a thickness of 4 mm and annealed. Samples were cold rolled 30%, 60%, 80% and 90% from the initial thickness in successive steps in a laboratory rolling mill. After cold rolling, specimens were annealed at temperatures of 955, 985 and 1010°C and then water cooled.

A Philips XPRO X-ray diffractometer with Co–Kα radiation was used to measure three incomplete pole figures (110), (200) and (211) with a maximum tilt of 85° on the central layer of the sheet. Orientation distribution functions (ODF) were calculated for these three pole figures using a series expansion method up to $L=22$ with the software POPLA.

An Oxford Crystal 300 EBSD system attached to a Philips XL-30 scanning electron microscope was used for microtexture, phase and recrystallization analyses. Precipitates were analyzed with an Atomic Force Microscope.

3. Results and discussion

Fig. 1 shows a schematic of the $\varphi_2=45°$ Bunge ODF section that contains the texture components discussed in this work. In this representation, the intensity $f(g)$ is plotted against the Euler angles φ_1 and ϕ.

Fig. 2 presents Bunge ODF sections at $\varphi_2=45°$ for samples cold rolled 30%, 60%, 80% and 90%. The deformation texture increases in intensity with increasing plastic deformation. The 30% deformed sample is characterized by two fibres (111) and (001). With increasing deformation to 60% in thickness the intensities {111} <121> and {001} <110> become the most intense. Continuing the deformation process to 80% in thickness the {001} <110> components become more intense and {111} <121> moves to {111} <011>. The sample deformed 90% exhibits a very intense {001}

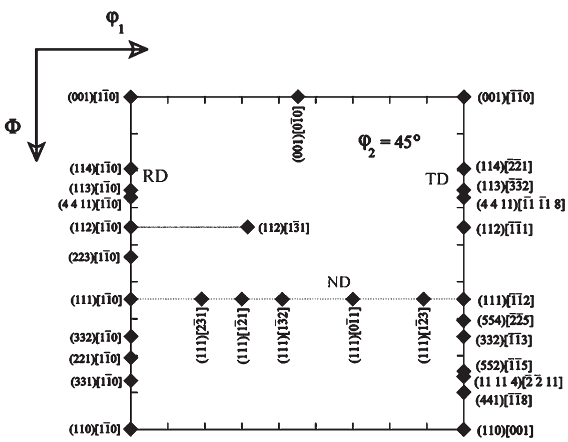

Fig. 1. Section for $\varphi_2=45°$ showing the position of main orientations along with the RD, TD and ND.

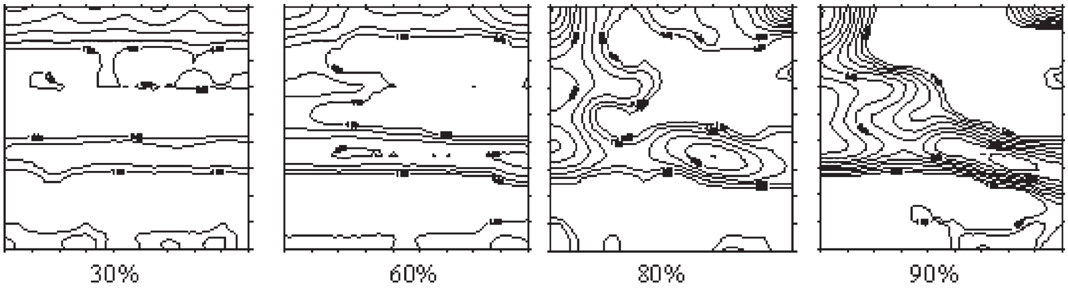

Fig. 2. Bunge ODF sections for samples cold rolled 30%, 60%, 80% and 90% in thickness.

<110> texture component and in the {111} plane the {111} <011> moves to a position between {111} <011> and {111} <123>. This texture is typical of cold rolled bcc material [6].

Fig. 3 shows the evolution of texture of cold rolled samples after annealing. This texture behavior is typical of recrystallized bcc-steel. It can be observed a decreasing of the α fibre orientation and an increase of the rolling texture component {111} <112>. Samples annealed at 1010 °C show no component in plane (100) and the main texture component is {111} <112>. The rolling texture component {001} <110> rotates to directions close to {110} <100>, the Goss texture.

Table 2 shows calculated values for r_m and Δr for samples cold rolled and annealed at the three different temperatures. The condition that results in the best deep drawing operation is that with the highest r_m value and

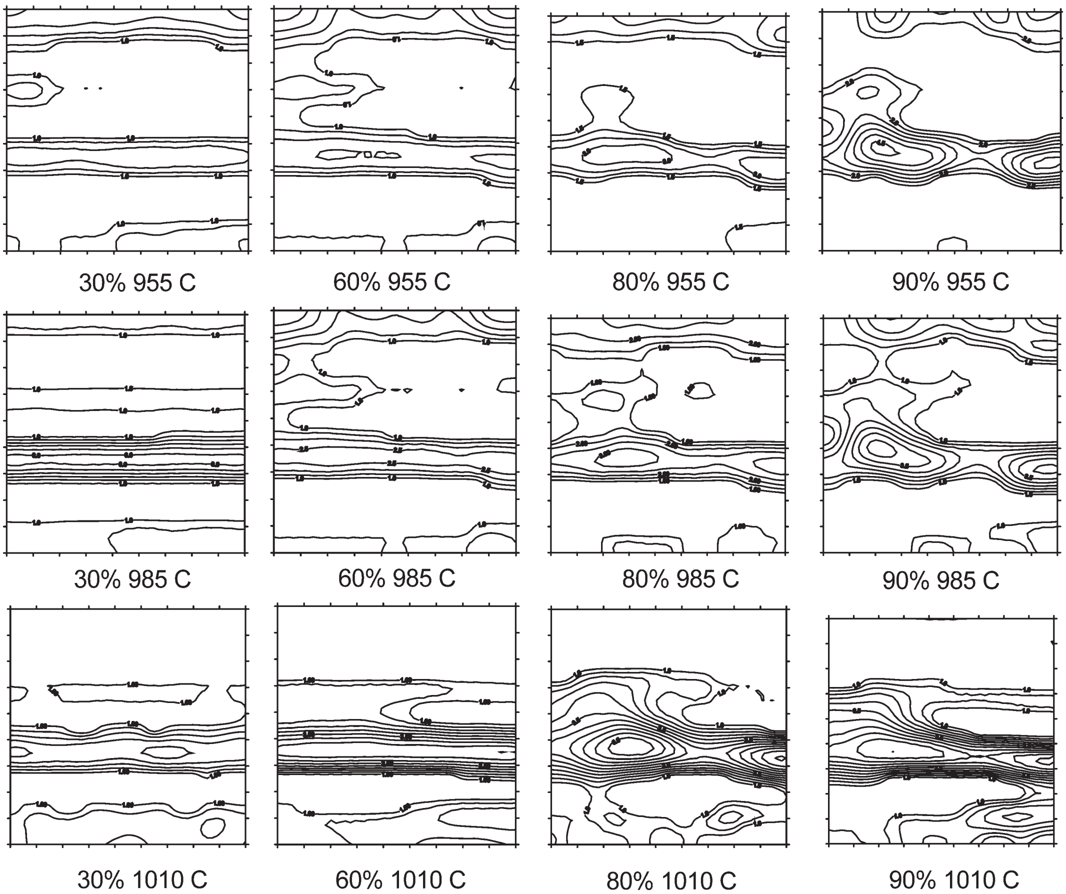

Fig. 3. Bunge ODF φ_2=45° sections for samples cold rolled 30%, 60%, 80% and 90% in thickness and annealed at 955, 985 and 1010 °C.

Table 2
Values for r_m and Δr

	ST		955 °C		985 °C		1010 °C	
	r_m	Δr	r_m	Δr	r_m	Δr	r_m	Δr
30%	0.63	−0.005	0.65	−0.130	0.82	0.020	1.29	0.060
60%	0.70	−0.060	0.70	−0.060	0.70	−0.060	1.69	0.040
80%	0.73	−0.285	0.68	−0.005	0.67	−0.015	1.71	0.085
90%	1.08	−0.570	0.78	−0.230	0.71	−0.040	1.93	−0.200

Δr values close to zero. The samples deformed 30%, 60% and 80%, annealed at 1010 °C, presented the highest values for r_m and Δr values closer to zero. In addition to this, the samples deformed 30% and 60%, annealed at 1010 °C, show the required texture for deep-drawing i.e., a {111} fibre texture parallel to normal direction.

Electron back scattering diffraction (EBSD) was used to analyze grain size variation. EBSD can be regarded as a reference method for measuring grain size. It not only enables each grain to be examined, but it also enables this to be accomplished according to the misorientation angle between two grains. Fig. 4 compares the grain size for samples cold rolled 30% and 60% in thickness and annealed at different temperatures. The grain size obtained for samples cold rolled 30% and annealed at 955, 985 and 1010 °C was 43.0 μm (ASTM number 7). Samples cold rolled 60% and annealed at 955, 985 and 1010 °C exhibited a grain size of 31.0 μm (ASTM number 8). No variation of grain size was observed as a function of annealing temperature. An increase in reduction from 30% to 60% in thickness reduced the grain size.

Fig. 5(A) and (B) presents optical microscopy images of the sample cold rolled 60% and annealed at 955 °C. Coarse TiN square precipitates are observed. Intragranular particles are also observed in Fig. 5(B). More detailed images of these particles

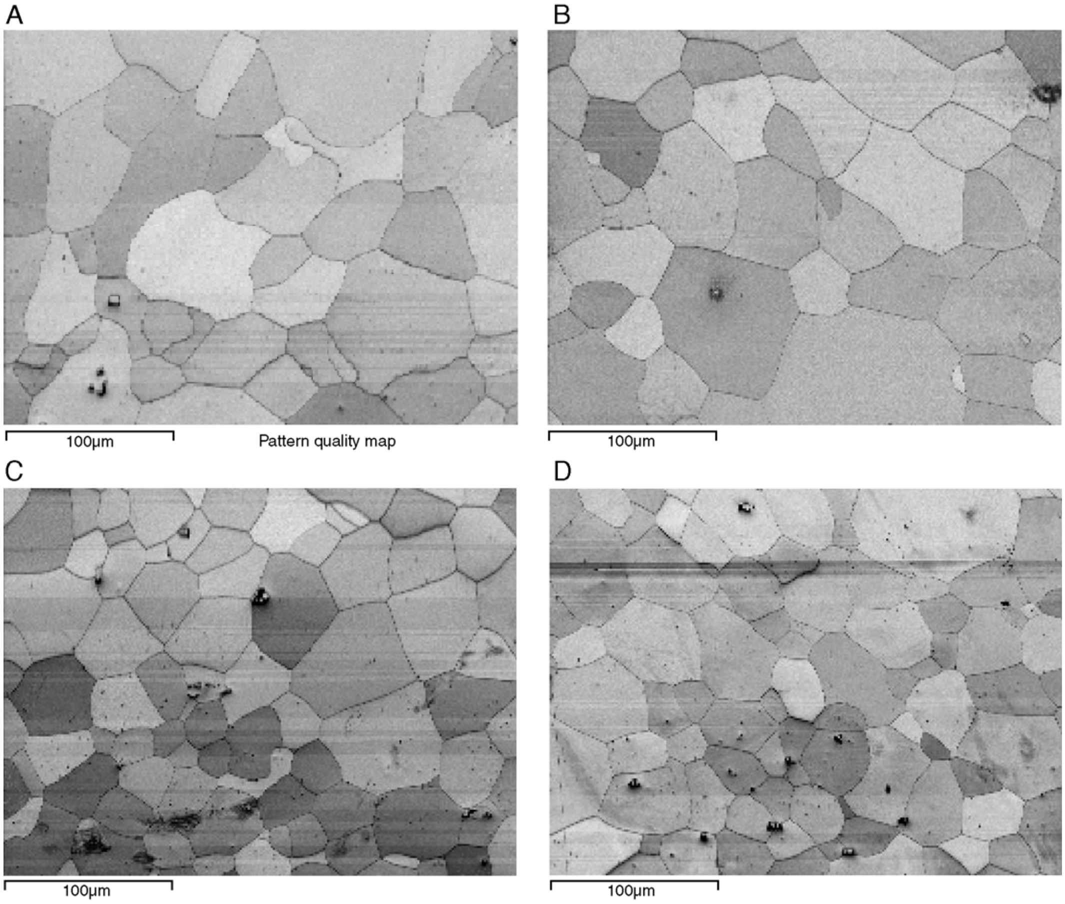

Fig. 4. Pattern quality maps for samples cold rolled 30% and annealed at (A) 955 and (B) 1010 °C and for samples cold rolled 60% and annealed at (C) 955 and (D) 1010 °C.

were obtained by atomic force microscopy (Fig. 6A and B). They are likely hexagonal carbides or nitrides of the stabilizing elements present in the steel (Nb or Ti).

4. Conclusions

Samples cold rolled 30% and 60% and annealed at 1010 °C presented microstructures, textures and mean *r* or Lankford values most suitable for deep-drawing operations. Samples annealed at 1010 °C presented no component in plane (100) and the main texture component was {111} <112>. They also presented some Goss texture, {110} <100>. An increase of the annealing temperature from 955 to 1010 °C did not influence grain size. The increase of deformation decreases the grain size after annealing. Analysis of the microstructure indicated the presence of some carbides and nitrides, probably those of Nb and Ti.

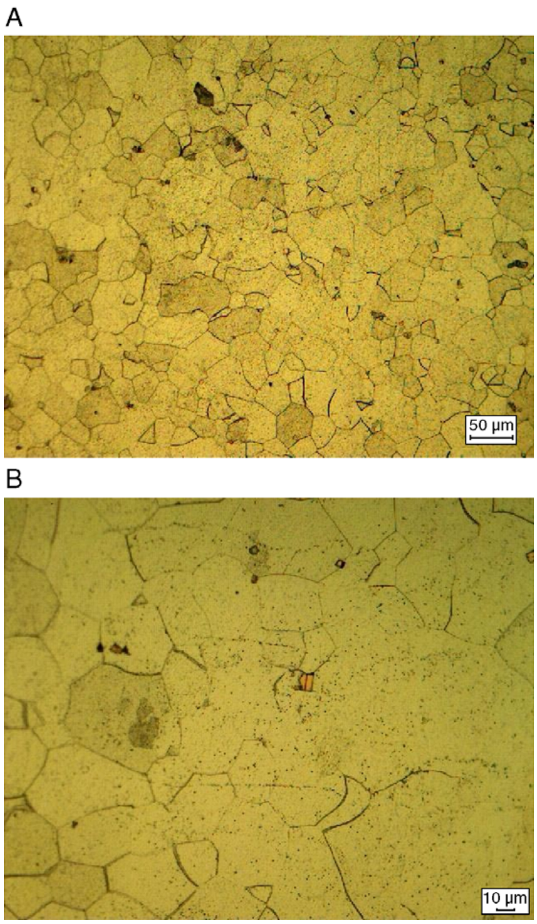

Fig. 5. Microstructure of a sample cold rolled 60% and annealed at 955 °C (A) 200 × (B) 500 ×.

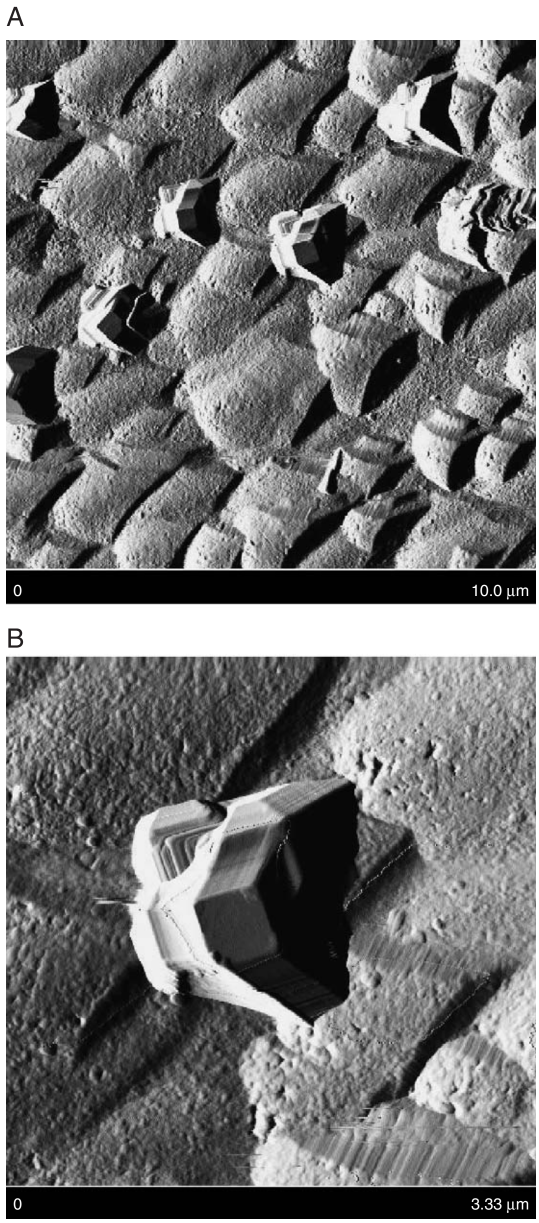

Fig. 6. Aspect of precipitates obtained by AFM.

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