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The behavior of a 1.4301 stainless steel subjected to cryogenic temperatures

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Abstract

Usually equipments that work at low and cryogenic temperatures are made of stainless steel due to their good mechanical and anti corrosion properties. The best stainless steels for this kind of applications are the ones that present an austenitic microstructure. The austenitic microstructure is the most plastic phase of the $Fe\text{-}Fe_3C$ alloys but this gives it a very good resilience at low temperature. This paper analysis the behavior of a 1.4301 stainless steel subjected to prolonged exposure to cryogenic temperatures and thermal cycles of cooling to cryogenic temperatures and heating to room temperature. The cryogenic temperatures were obtained by immersing the samples in liquid air which has a temperature of -196 ºC. The samples were analyzed using scanning electron microscopy, optical microscopy and x-ray diffraction.

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Keywords: stainless steel; cryogenic temperatures; SEM; XRD.

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1. Introduction

The materials used in the construction of machine parts and equipments which are subjected to low temperatures in their normal functioning regime are usually stainless steels. At temperatures lower then -40 ºC the majority of

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stainless steels loses their mechanical properties and become brittle [1]. For this reason at temperatures lower then -40 ºC stainless steels with austenitic microstructure are used.

Usual carbon steels have at room temperature a microstructure composed of alpha ferrite and cementite. Alpha ferrite is a solid solution of carbon dissolved in alpha iron which solidified in a body centered cubic lattice. The austenitic phase can exist in carbon steels at temperatures higher then 727 ºC. The austenitic phase is composed of carbon dissolved in gamma iron and solidified in face centered cubic lattice. To stabilize the austenitic phase at room temperature the steel is alloyed with nickel (18 – 20 %) and to give the steel stainless properties it is additional alloyed with chromium [3].

One characteristic of stainless steels with austenitic microstructure is that they contain grains with annealing twins. The annealing twins are portions of grains that have a different orientation then the rest of the crystal and they are a plastic deformation mechanism which occurs at grain level. The mechanism consists of a translation between two parallel planes of the lattice between each other such that the deformed portion of the crystal becomes symmetric to the undeformed portion of the crystal.

This paper analyses the behavior of a 1.4301 stainless steel subjected to prolonged exposure to cryogenic temperatures and to thermal cycles of cooling at cryogenic temperatures and heating to room temperature.

2. Experimental procedure

The samples used in analyses were made of a hot rolled 1.4301 stainless steel. To reach cryogenic temperatures, the samples were immersed in liquid air which has a temperature of - 196 ºC. One of the samples was kept immersed in liquid air for a period of 48 hours to see the effect of prolonged exposure to cryogenic temperatures. The second sample was subjected to thermal cycles of cooling in liquid air until it reached thermal equilibrium followed by rapid heating in water at room temperature. The sample was subjected to 35 cycles of cooling – heating.

After the test, the samples were analyzed using scanning electron microscopy (Quanta 200 3D microscope, working in High Vacuum module), optical microscopy (Leica DMI 5000 M) and X-ray diffraction (Panalytical X'Pert Pro MRD). The chemical composition of the 1.4301 stainless steel is: Fe 68,73%, C 1,18%, O 2,37%, Si 0,82%, Cr 18,53% and Ni 8,37%. Before subjecting the samples to cryogenic temperatures they were cut and polished and after immersion in liquid air they were cleaned with a special solution in an ultrasonic bath and etched with 4% Picral using heat. The purpose of the SEM and optical analyses was to determine the micro structural changes that appeared in the samples due to the test and the XRD analyses highlighted the phase changes in the material.

3. The results of the tests

3.1. The micro structural and phase characterization of the 1.4301 material from which the samples were made of

The first analyzed sample was not subjected to cryogenic temperatures and was used for comparison to the other two samples.

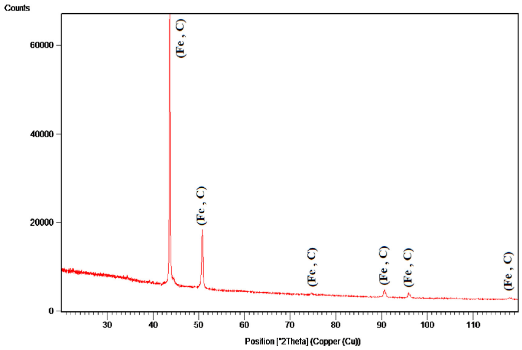

Fig. 1. The X-ray diffraction pattern for the sample not subjected to cryogenic temperatures

Fig. 1 show the results obtained from the XRD analyses made on the sample not subjected to cryogenic temperatures. The analyses show that the material is made of only from one phase. The intensity picks at different position angles indicate that in the material only the austenitic phase is present.

The Miller indices which characterize the crystallographic planes of the austenitic phase in the material are showed in Fig. 2.

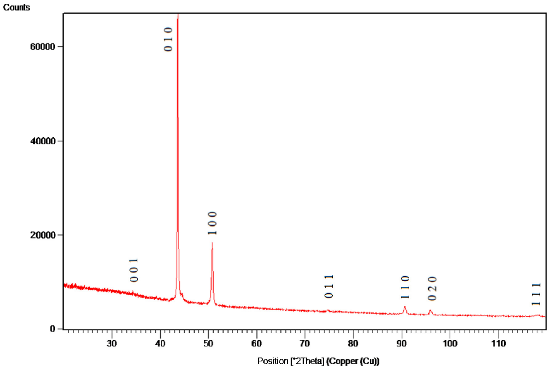

Fig. 2. The Miller indices for the sample not subjected to cryogenic temperatures

The crystallographic parameters of the elementary lattice are showed in Table 1. The parameters are specific for the austenitic phase which crystallizes in a face centered cubic lattice.

Table 1. The crystallographic parameters of the elementary lattice for the austenitic phase

Parameters	Fe,C - Cubic
a (Å)	3,6000
b (Å)	3,6000
c (Å)	3,6000
Alfa (°)	90,0000
Beta (°)	90,0000
Gama (°)	90,0000

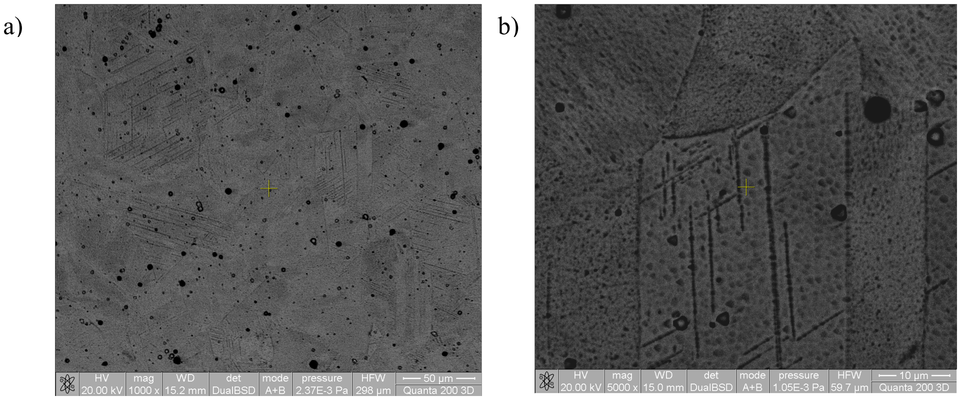

Fig. 3. SEM images of the sample not subjected to cryogenic temperatures: (a) at a magnitude of 1000x; (b) at a magnitude of 5000x

The SEM images in Fig. 3. (Fig. 3. (a) at a magnitude of 1000x and Fig. 3. (b) at a magnitude of 5000x) show that the stainless steel material is made of two types of grains. This fact is also highlighted in the optical analyses from Fig. 4 (Fig. 4. (a) at a magnitude of 100x and Fig. 4.(b) at a magnitude of 500x).

The grains without annealing twins have a polyhedral shape and they are colored white in the optical analyses. The grains with annealing twins show a more pronounced profile then the other grains and they have only a few twining planes.

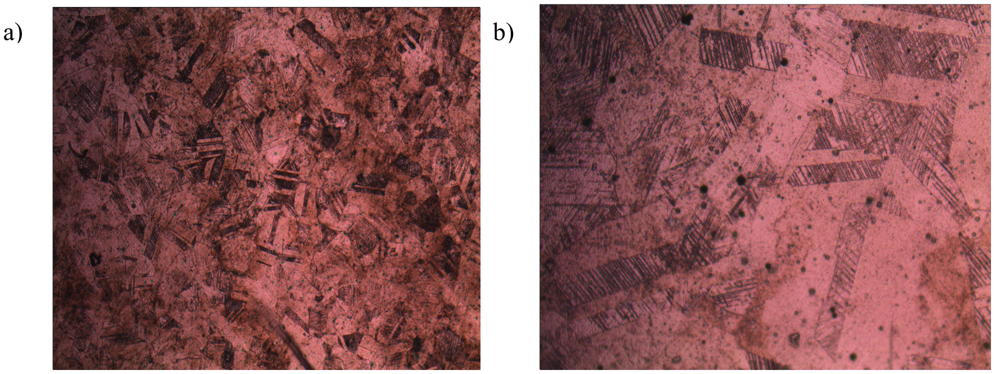

Fig. 4. Optical microscopy images of the sample not subjected to cryogenic temperatures: (a) at a magnitude of 100x; (b) at a magnitude of 500x

3.2. The results of the sample subjected to cryogenic temperatures in thermal cycles of cooling-heating

The sample subjected to 35 thermal cycles of cooling – heating shows no phase changes in comparison to the previous presented results. The material is still composed of a single austenitic phase, fact highlighted by the XRD analysis result showed in Fig. 5.

The morphology of the samples micro structure changes due to the thermal cycles as can be observed in the SEM analysis images from Fig. 6. The dimension of the grains with annealing twins increased and the number of twinning planes also increased inside the grains. The twinning planes have preferential directions corresponding to the thermal expansions and contractions that occur in the material during cooling and heating. The twinning planes can facilitate plastic deformation in the material in directions tangent to the planes. This fact leads to anisotropy regarding the tensile yield stress of the material [2].

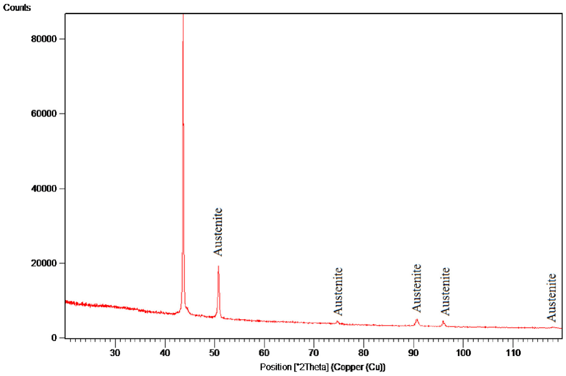

Fig. 5. The X-ray diffraction pattern for the sample subjected to cryogenic temperatures in thermal cycles of cooling - heating

The optical analysis (presented) shows that during the test some grains with annealing twins oriented in the direction of the thermal gradient that appeared in the material during the cycles of cooling – heating. This fact increases the anisotropy of the mechanical properties in the material.

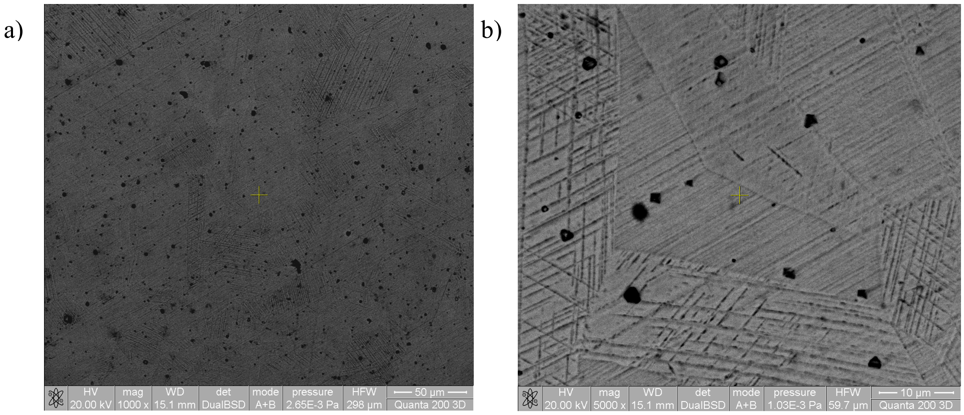

Fig. 6. SEM images of the sample subjected to cryogenic temperatures in thermal cycles of cooling - heating: (a) at a magnitude of 1000x; (b) at a magnitude of 5000x

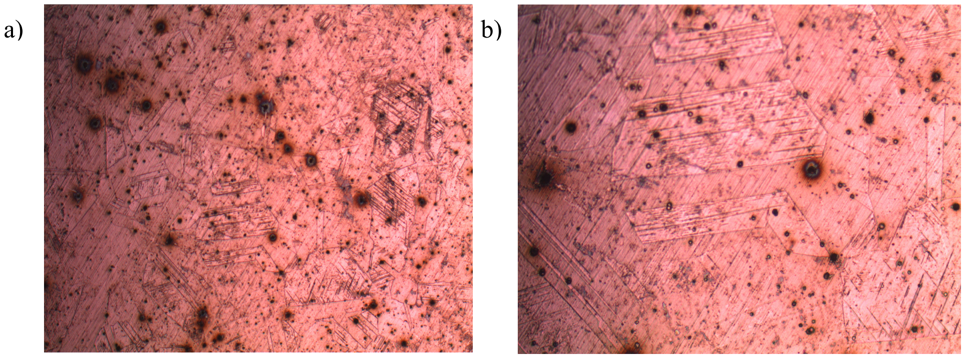

Fig. 7. Optical microscopy images of the sample subjected to cryogenic temperatures in thermal cycles of cooling - heating: (a) at a magnitude of 100x; (b) at a magnitude of 500x

3.3. *The results of the sample subjected to prolonged exposure to cryogenic temperatures*

The last sample analyzed is the one immersed for 48 hours in liquid air. The XRD analysis presented in Fig. 8 show that the austenitic phase dissociated due to prolonged exposure to cryogenic temperatures and a new phase of Fe-Cr alloy formed.

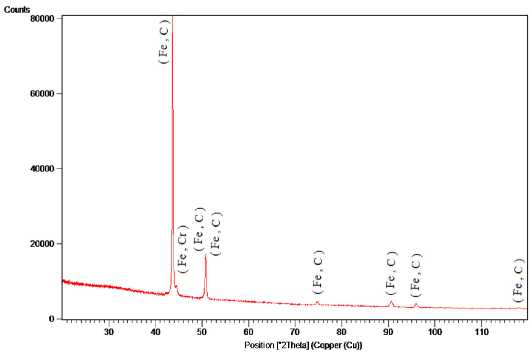

Fig. 8. The XRD analyses results for the sample subjected to prolonged exposure to cryogenic temperatures

The Miller indices (the (2 0 2) indices of the Fe-Cr phase) presented in Fig. 9 indicate that the new formed phase consists of a complex phase that has a chromium atom inside the face centered cubic lattice of gamma iron which centers the lattice.

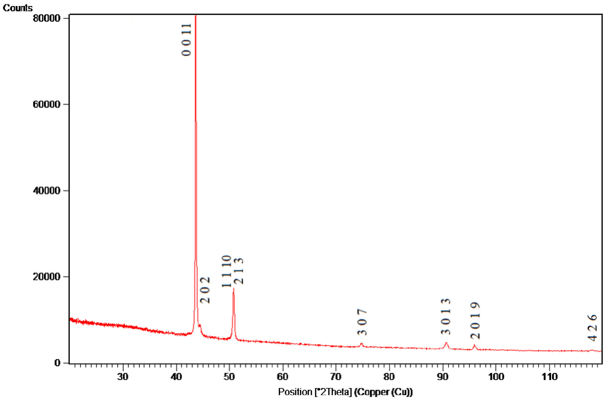

Fig. 9. The Miller indices for the sample subjected to prolonged exposure to cryogenic temperatures

In Table 2 are presented the crystallographic parameters of the lattice for the two phases that appeared in the sample subjected to cryogenic temperatures.

Table 2. The crystallographic parameters of the elementary lattice for the two phases of the sample subjected to prolonged exposure to cryogenic temperatures

Parametri	Fe,C - Cubic	Fe_Cr - Cubic
a (Å)	3,6000	2,8760
b (Å)	3,6000	2,8760
c (Å)	3,6000	2,8760
Alfa (°)	90,0000	90,0000
Beta (°)	90,0000	90,0000
Gama (°)	90,0000	90,0000

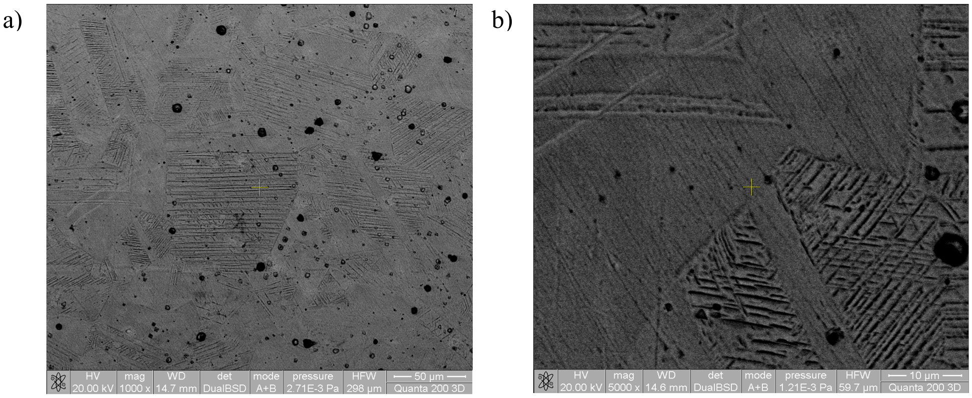

Fig. 10. SEM images of the sample subjected to prolonged exposure to cryogenic temperatures: (a) at a magnitude of 1000x; (b) at a magnitude of 5000x

The SEM analysis of the sample, presented in Fig. 10, shows an even more accentuated increase in dimensions of the grains with annealing twins and also the increase of the number of twinning planes in the grains. The planes of the annealing twins are oriented in two directions, directions on which the material will plastically deform more easily. The increase of the grains with annealing twins is also showed in the optical analysis from Fig. 11. The grains with annealing twins are completely black after this test due to the high number of twinning planes.

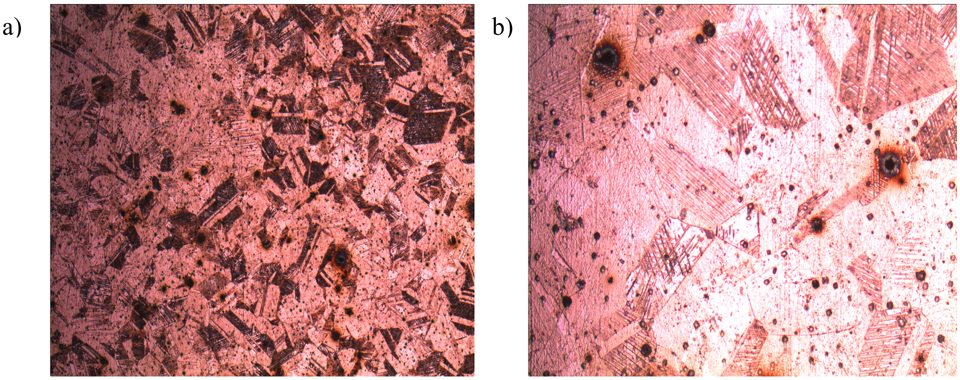

Fig. 11. Optical microscopy images of the sample subjected to prolonged exposure to cryogenic temperatures: (a) at a magnitude of 100x; (b) at a magnitude of 500x

4. Conclusions

By subjecting the stainless steel to thermal cycles of cooling-heating its micro structure changes causing an increase of the dimensions of the grains with annealing twins and also increases the number of twinning planes inside the grains. Also due to the thermal expansion and contraction of the material in certain directions the twinning planes have preferential directions. The thermal gradient inside the material leads to the orientation of the grains with annealing twins in the direction of the gradient. The prolonged exposure to cryogenic temperatures leads to an even greater expansion of the grains with annealing twins and orients the twinning planes in two directions. Also the number of twinning planes increases inside grains even more then in the case of the sample subjected to thermal cycles. Another change that occurs with this sample is that the austenitic phase dissociates and another phase of Fe-Cr forms which can lead to the loss of mechanical and anti corrosion properties of the material. The changes that appear in the stainless steel due to exposure to cryogenic temperatures can lead to preferential directions of plastic deformation and anisotropy of the mechanical properties of the material.

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