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# ASPECTS CONCERNING THE CRYSTAL LATTICE BEHAVIOR OF AUSTENITIC STEEL AT LOW TEMPERATURE

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**Abstract:** Two austenitic stainless steels for investigation under cryogenic condition were considered. Both X8CrNi18-12 and X12CrNi25-21 were investigated by X-ray diffraction at several cryogenic temperatures of 298 K, 223 K, 173 K, and 123 K. The crystal parameter and thermal linear expansion coefficient were calculated from the X-ray diffraction pattern. The major observation was the increasing of diffraction angles as temperature decreases. The X8CrNi18-12 crystal parameter presents a significant decreasing related to the same condition applied to X12CrNi25-21. We observe the same tendency for thermal linear expansion coefficient variation which means that X8CrNi18-12 becomes harder and more brittle than X12CrNi25-21 in the same cryogenic condition. Finally the X8CrNi18-12 proves to be useful for cryogenic applications which need high hardness instead of a good toughness and X12CrNi25-21 prove to be useful for cryogenic applications which need a better toughness. **Key words:** X-ray diffraction, austenitic stainless steels, elementary cell.

#### **1. INTRODUCTION**

The cryogenic behaviour investigation of materials represents a very important research field in order to improve the materials quality for a various technological and industrial materials aplications. Cryogenic researches were started long time ago [1], continuing to be of interest for applications such special magnets development and special composite materials [2, 3] for a wide range of applications from aircrafts to food industry. Many stainless steels are used for special cryogenic application, for example the X8CrNi18-12 stainless steel is also accepted to be in contact with food [4], resulting a special interest for the food industry.

Cryogenic procedures are needed sometimes even at foods preparation. A very useful method for the stainless steel cryogenic behavior is the X – ray diffraction, performed under nitrogen cooling [5, 6]. It is expected that the severe temperature decreasing to attend cryogenic state affects the intimate structure of steels having a great influence to the mechanical properties of manufactured pieces.

The aim of this paper is to figure out some characteristics of cryogenic behavior of two austenitic stainless steels X8CrNi18-12 and X12CrNi25-21.

#### **2. THEORETICAL TREATMENT**

The X-ray diffraction investigation is able to reveal the crystalline state - the crystal structure. Furthermore, the lattice parameter could be calculated by the least square method

$$\mathbf{a}_{i} = \mathbf{a}_{0} + \mathbf{b}\mathbf{f}_{i},\tag{1}$$

where:  $a_0$  – extrapolated lattice parameter at  $2\theta$ =  $180^{\circ}$  (minimum error of a),  $a_i$  – resulted lattice parameter for each diffraction peak,  $f_i$  – extrapolation function at  $\theta_i$  [6]

$$f = \frac{\cos^2 \theta}{\sin \theta} + \frac{\cos^2 \theta}{\theta}.$$
 (2)

The thermal linear expansion coefficient (TLEC) is a very important physical and technological value. Conssidering this, we calculate the thermal liniar expansion coefficients by equation

$$\alpha = \frac{2\frac{(a_{01} - a_{02})}{(a_{01} + a_{02})}}{(T_1 - T_2)},$$
(3)

where:  $\alpha$  is the TLC,  $a_{01}$  – the crystal parameter at temperature  $T_1$  and  $a_{02}$  – the crystal parameter at temperature  $T_2$ .

## **3. EXPERIMENTAL PROCEDURE**

Samples of X8CrNi18-12 and X12CrNi25-21 austenitic stainless steels were cut from rolled plates. The samples were investigated at cryogenic temperatures on a DRON 3 diffractometer equipped with low temperature URNT-180 device,  $Cu_{k\alpha}$  radiation was filtered with a Fe filter of 20 µm thickness, analysis was performed on a range between: 40 - 98 20 degree. The X-ray investigation was done for several low temperatures of: 298 K, 223 K, 173 K, and 123 K.

#### 4. RESULTS AND DISCUSSION

The X-ray diffraction spectra for X8CrNi18-12 and X12CrNi25-21 stainless steels are presented in Figure 1 and, respectively, Figure 2. We notice that the spectra at room temperature are almost identical for both samples, Figures 1a and 2a.

There are featured austenite peaks and some residual bronze peaks from the sample support.



Fig. 1. X-ray diffraction patterns for X8CrNi18-12: a) 298 K , b) 223 K, c) 173 K, and d) 123 K.  $\triangleright$  = bronze sample holder.



Fig. 2. X-ray diffraction patterns for the X12CrNi25-21: a) 298 K, b) 173 K, and c) 123 K.

The temperature decreasing affects the shape of austenite diffraction peaks by reducing of their height and of the full width at the half height FWHM and the increasing of diffraction angle  $2\theta$ , as a consequence of decreasing of crystal parameter for both samples, mainly for X8CrNi18-12 where the peaks variation is more intense.

The crystal parameters  $a_i$  were calculated from the X-ray diffraction according to FCC indexation for each peak. The calculation of crystal parameter  $a_0$  was done by the least square method, equations (1) and (2).

The value of  $a_0$  parameter represents the reference unit of austenite FCC lattice being the most important parameter to be observed. It is presented the variation of  $a_0$  for X8CrNi18-12 in Figure 3 and  $a_0$  for X12CrNi25-21 is in Figure 4.



Fig. 3. Austenite FCC crystal parameter evolution versus measuring temperature for X8CrNi18-12.

The variation in Figure 3, shows a continuous tendency of crystal parameter decreasing as temperature decreases, meaning an intensive shrinkage of the crystal lattice,

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proportional to the decreasing of temperature. This behavior is to affect the dimensional and maybe shape aspect of the steel part designed to work under cryogenic condition.

The decreasing tendency of  $a_0$  is also continuous for X12CrNi25-21, Figure 4, but it decreases slowly than for X8CrNi18-12, Figure 3.



Fig. 4. Austenite FCC crystal parameter evolution versus measuring temperature for X12CrNi25-21.

This slowly decreasing of crystal parameter of X12CrNi25-21 compared to X8CrNi18-12 proves a more thermal stability of the richest Cr, Ni alloyed stainless steel. The fact observed in crystal parameter versus temperature variation equations

$$\begin{cases} a_{0 X8CrNi18-12} = 0,0068T + 356,72; \\ a_{0 X12CrNi25-21} = 0,0107T + 357,31. \end{cases}$$
(4)

The cooling cycles to the cryogenic temperatures and again to the room temperature could stress the material being a potential damage promoter to the inside of steel cristallites.



Fig. 5. Thermal liniar expansion coefficient versus temperature for X8CrNi18-12.

So a very important physiscal and technological value is the thermal linear expansion coefficient (TLEC). Conssidering these we calculate the TLEC's by equation (3), and temperature dependence of  $a_0$  by (4).

The resulted values of TLEC for X8CrNi18-12 are presented in Figure 5 and for X12CrNi25-21 in Figure 6.



Fig. 6. Thermal liniar expansion coefficient versus temperature for X12CrNi25-21.

In Figure 5 arround 150 K we observe a smaller value of TLEC compared to average, but shortly arround to 200 K its value overtake the average and the increassing tendency continue slowly to the room temperature. This evolution proves the nonliniarity in TLEC behavior during cryogenic measurement.

Similar aspect is featured for X12CrNi25-21 in Figure 6, but the values of TLEC are considerably smaller then those resulted for X8CrNi18-12. The less values of TLEC prove a high thermal stability of richest Co and Ni stainless steel.

We found in literature mentioned that the fracture elongation and resilience are considerable decreased by cryogenic treatment [7], while the strength properties such  $R_{p0.2}$  and  $R_m$  are increased than the values for room temperature.

That technical observations are proved by the severe reducing of crystal parameter and TLEC which conduct to a transition of material from a toughness one to a brittle one, the high values of  $R_{p0.2}$  and  $R_m$  combined with low resilience and almost none fracture elongation is a characteristic for brittle materials [8]. In this case it must to be count to that value in cryogenic working pieces design.

The observed behavior of crystal parameter and TLEC has serious implication in parts design and material use destination because of affecting the mechanical properties. The X8CrNi18-12 stainless steel prove to be hard and relatively brittle under cryogenic conditions 342

being able to be used in proper conditions to avoid cracks propagation or braking due to the brittle failure. At the same condition the X12CrNi25-21 prove to have more thermal stability and an increased toughness than previous one.

## **5. CONCLUSIONS**

stainless steels for Two austenitic investigation under cryogenic condition were investigated. Both X8CrNi18-12 and X12CrNi25-21 were investigated by X-ray diffraction at several cryogenic temperatures of: 298 K, 223 K, 173 K, and 123 K. The crystal parameter and thermal linear expansion coefficient (TLEC) were calculated from the obtained X-ray diffraction pattern. We observe the diffraction angles increase as that temperature decreases due to the shrinkage of crystal lattice. The X8CrNi18-12 crystal parameter presents a significant decreasing related to the same condition applied to the X12CrNi25-21. We observe the same tendency for thermal linear expansion coefficient variation which means that X8CrNi18-12 becomes harder and more brittle than X12CrNi25-21 the in same cryogenic condition. Finally the X8CrNi18-12 proves to be useful for cryogenic applications which need high hardness instead of a good toughness and

# X12CrNi25-21 prove to be useful for cryogenic applications which need a better toughness. **REFERENCES**

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## Aspecte privind comportamentul rețelei cristaline a unor oțeluri austenitice la temperaturi joase

**Rezumat:** Pentru prezentul studiu au fost luate in considerare două mărci de oțel inoxidabil austenitic. Ambele mărci de oțel, X8CrNi18-12 and X12CrNi25-21, au fost investigate prin difrație cu raze X la diferite temperature joase: 298 K, 223 K, 173 K, and 123 K. Din difractograme s-au calculat parametrul cristalografic și coeficientul de expansiune termică liniară. S-a observat creșterea sensibilă a valorii unghiurilor de difracție pe măsură ce temperatura scade. Parametrul cristalografic al X8CrNi18-12 prezintă o scădere mai pronunțată decât a X12CrNi25-21 în aceleași condiții ale scăderii temperaturii. Se observă o variație similară și pentru și coeficientul de expansiune termică liniară, ceea ce înseamnă că X8CrNi18-12 devine mai dur și mai fragil decât X12CrNi25-21 în aceleși condiții criogenice. În final rezultă că oțelul X8CrNi18-12 este util pentru aplicații criogenice ce necesită duritate ridicată și o tenacitate mai redusă în timp ce X12CrNi25-21 se pretează pentru aplicații criogenice ce necesită o tenacitate mai bună.

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