TECHNICAL UNIVERSITY OF CLUJ-NAPOCA

ACTA TECHNICA NAPOCENSIS

Series: Applied Mathematics, Mechanics, and Engineering
Vol. 65, Issue I, March, 2022

LOW TEMPERATURE STRUCTURE OF SOME METALS AND ALLOYS

Vasile-Marius BĂRĂIAN, George ARGHIR

Abstract: *Experimental research reveals that most metallic materials generally show in the range of low temperatures (+15 ... -153 °C) the reduction of the vibration amplitude of the nodes of the crystalline lattice, a fact reflected in the X-ray diffractograms; the bottom is reduced and the picks are taller and narrower. The parameter of the crystal lattice decreases with the decrease of the temperature, the density increases, the elementary volume of the crystal lattice decreases. As the temperature decreases, phase transformations take place in special steels by transforming the residual austenite into martensite. The same phenomena occur in lead and tin: the crystallographic parameter decreases, the coefficient of thermal expansion shows a jump, the density increases, the volume of the elementary cell decreases with the decrease of the temperature. The transformation of white into gray tin is not noticeable.*
Key words: *Crystalline structure. Low temperature, structural transformation.*

1. INTRODUCTION

The study of the structural and physical properties of materials, in general, of some metals and alloys in particular, at low temperatures (+15 ... -153 ºC) is a field of great relevance, now that the cold technique is gaining current applications in both industrial, as well as in aviation, space and other areas where temperatures close to the liquefaction temperature of nitrogen

In the field of low temperatures, in some metals and alloys there may be phase transformations, changes in the coefficient of thermal expansion, changes in the heat transfer coefficient, possible changes in the sliding planes that can lead to superplasticity [1].

Some of the modern methods used in the treatment of metals and their alloys is tempering at low temperatures. While the science of heat treatment is well known and widely understood, the principles of low temperature tempering remain a mystery to many people working in the industry. Thus, the information about this process is full of contradictions and unanswered questions. Until recently, tempering at low temperatures was considered worthless due to the property of most materials to become brittle at these temperatures [2, 3].

Heat treatment at low temperatures is beginning to prevail due to the superior properties; it improvs the wear resistance. It has been found that through cryogenic cooling, the durability of the parts increases by about 25 %, which represents lower costs and saves time required to replace worn parts [4].

Regarding the X-ray diffraction research at low temperatures, they have been done for a long time, but the works published in the field are very few, these researches being fields of advanced technique, of last moment [2].

In the face of the lowest possible production costs, in the rigors of fierce competition, which the development of the 21st century generates, the study of the structural and physical properties of materials at low temperatures is of more than stringent practical importance [5].

2. EXPERIMENTAL FACILITY

In order to highlight the structural changes that occur at low temperatures, experimental research was performed in the diffractometry laboratory of the Faculty of Materials and

Received: 03.01.22; **Similarities**: 28.02.22: **Reviewed**: 21.02./28.02.22: **Accepted**:22.03.22.

Environmental Engineering of the Technical University of Cluj-Napoca, using the X-ray diffractometer DRON-3, on which mounted the Roentgen plant at low temperatures URNT – 180, the cryogenic liquid (liquid nitrogen) being transferred from the Dewar storage vessel to the working plant by means of a transfer device.

The following metals and alloys: iron, 600-3U non-alloy cast iron (EN-GJS), X3CrSi12-2 alloy cast iron (EN-GJL), C35R steel EN-10083), austenitic steels X3CrNi18-10 (SR EN 10088 / I), X8CrNi18-12 (SR EN 10088 / I) and X12CrNi25-21 (SR EN 10088 / I), ferritic-martensitic steel X20Cr13 (SR EN 10088 / I), HS 18-1-1 high speed steel, X200Cr12 ledeburite steel (SR EN 10088 / I), RUL1 bearing steel, lead and tin, were subjected to X-ray diffraction at different temperatures: 298 K (25 ° C, room temperature), 273 K (0 ° C), 223 K (-50 ° C), 173 K (-100 ° C). , 123 K (-150 ° C) and 113 K (- 160 ° C) during the research [1].

The measurement temperature of the samples was reached in the nitrogen vapor measuring space in the URNT-180 device. The device was placed and fixed on the X-ray diffractometer. Prior to the start of each measurement, the sample was kept at the measurement temperature for 10 minutes. The whole experimental facility is shown in Figure 1.

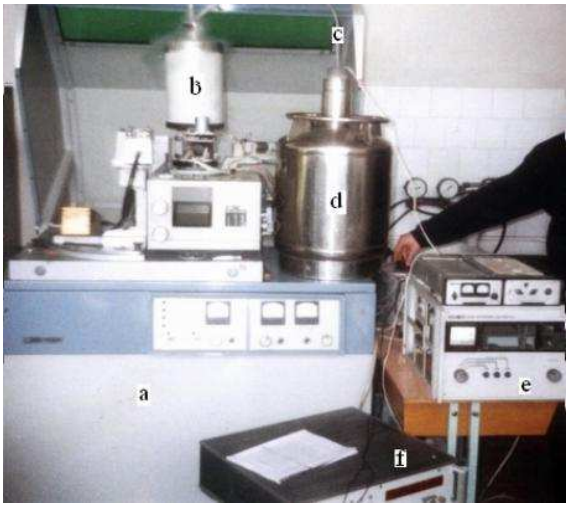

Fig.1. Experimental facility: a - DRON-3 diffractometer, b - Roentgen low temperature device URNT-180, c - nitrogen liquid transfer device, d - Dewar vessel (liquid nitrogen), e - control unit and control, f - voltmeter.

In order to detect the solid-state phase transformations that occur in high-alloy steels after hardening, followed by cryogenic cooling, microphotographs were made using the Neophot-2 optical microscope, as well as measurements of hardness and microhardness.

3. EXPERIMENTAL RESEARCH

Iron powder subjected to X-ray diffraction at temperatures of 298 K (25 ºC), 223 K (-50 ºC), 173 K (-100 ºC) and 123 K (-150 ºC) gives crystallographic parameters of 286.34, 286.02, 285.81 and 285.65 pm. The crystallographic parameters are in accordance with those published in the literature for room temperature [2]. With the reduction of the temperature, the decrease of the crystallographic parameter is noticed, the structure being ferritic (from indexing). The thermal shrinkage coefficient is not constant over the considered temperature ranges. The density of the iron increases with the decrease of the temperature, in the studied fields, and the volume of the elementary cell, as expected, decreases due to the thermal contraction.

600-3U non-alloy cast iron, X3CrSi12-2 alloy cast iron, C35R steel, X3CrNi18-10 austenitic steels, X8CrNi18-12, X12CrNi25-21, X20Cr13 ferrite-martensitic steel generally have the same characteristics as iron. The determined values (crystallographic parameters and coefficients of linear thermal expansion) are in accordance with the data in the literature [3].

Modification of the structure as a result of heat and cryogenic treatment applied to high-alloy steels revealed that HS 18-1-1 high speed steel: in annealed state, hardened and after cryogenic treatment shows changes in hardness (291, 856, 897 HV) and microhardness (252, 1570 and 1987 HV0.02). X-ray diffraction identified allied ferrite, allied austenite and allied martensite at the exposed temperatures. In the obtained diffractograms, Figure 2, the presence of diffraction maxima peaks corresponding to ferrite (F), martensite (M), austenite (A) and tungsten, chromium and vanadium carbides can be noticed.

The microphotographs taken, Figures 3, 4 and 5, revealed the structure of the steel in the three states.

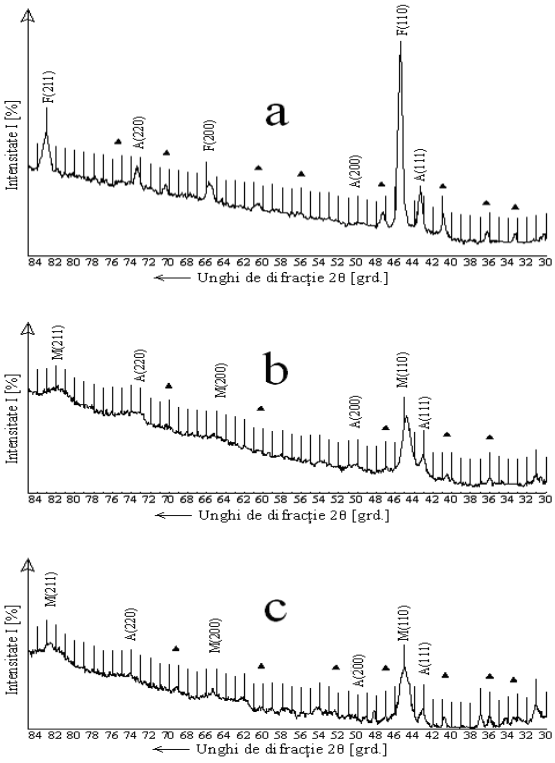

Fig. 2. HS 18-1-1 high speed steel diffractograms in annealed state (a) and after hardening (b) at 298 K and 123 K (c) respectively. F - allied ferrite, A – allied austenite, M – allied martensite, ▲ - Fe3W3C.

Figures 3 and 4 show that the formed martensite has a fine needle structure that it is not observed metallographically and, therefore, the structure appears to consist only of residual austenite and carbides.

The annealed X200Cr12 ledeburite steel was hardened, followed by cryogenic treatment. It has been observed that there have been changes with respect to the annealed state by hardening.

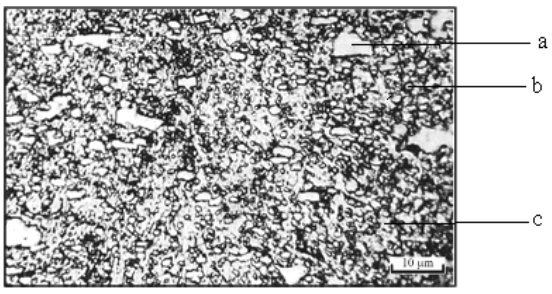

FIG. 3. Structure of annealed HS 18-1-1 high speed steel, chemical attack: royal water: a - primary carbide, b - secondary carbide, c - sorbitic eutectoid.

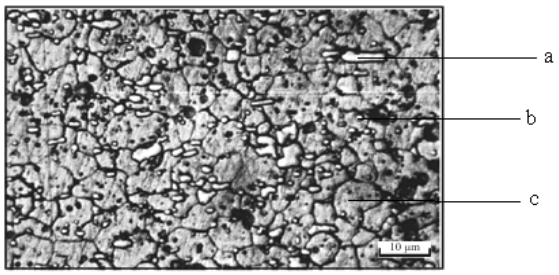

Fig. 4. HS 18-1-1 hardened steel structure in hardened state, chemical attack: royal water: a - primary carbide, b - secondary carbide, c - residual austenite.

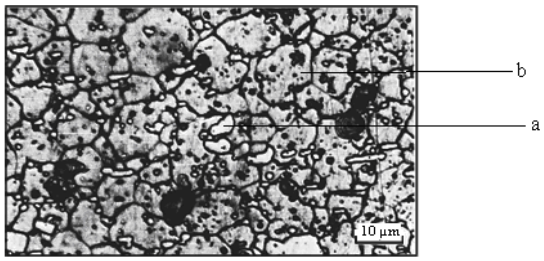

Fig. 5. HS 18-1-1 high speed steel structure after cooling to -150 ° C, chemical attack: royal water: a - carbide, b - residual austenite and martensite.

Microphotography, like X-ray diffraction and hardness measurements, led to the identification of phases: allied ferrite, allied austenite and allied martensite.

The crystallographic parameter was calculated for all cases and values comparable to those in the literature [5].

Microphotographs show the presence of primary and secondary carbides. The measured value of microhardness after cryogenic cooling was 1426 HV0.02, which means an increase of 9.7 % compared to that obtained after hardening, due to the transformation of a residual amount of residual austenite upon cooling.

RUL1 bearing steel is in annealed condition after hardening and cooled to 123 K: allied ferrite, allied austenite, allied martensite and chromium carbide, but in different proportions. The amount of residual austenite decreased by cooling to 6.44 % compared to 13.41 % in hardened steel, with a significant increase in microhardness to 1570 HV0.02, that is 34.5 % compared to hardened steel.

The beneficial effects of cooling are also shown on microphotographs; cooling increases the amount of martensite.

Lead subjected to X-ray diffraction at low temperatures shows a sharp decrease in the crystallographic parameter, the coefficient of thermal expansion [4] shows a jump in temperature, significantly increases the density, decreases the volume of the elementary cell. These data were also confirmed by the repetition of diffraction experiments.

Tin subjected to X-ray diffraction at low temperatures surprisingly showed that the transformation of white tin into gray tin does not occur even after a few days of maintenance at low temperatures; it maintained the structure of white tin, keeping its tetragonal structure centered in volume. The same general phenomenon of reduction of crystalline parameters (a_0, c_0) with the decrease of temperature is also noticed in tin, but the decrease is not linear, leading to variations of the tetragonality, density and volume of the elementary cell. These variations are attributed to possible trends of allotropic transformation to gray tin, delayed by the temperatures at which the sample was maintained.

4. CONCLUSION

Most metallic materials generally have a low temperature reduction in the vibration amplitude of the nodes of the crystal lattice, which is reflected in X-ray diffractograms.

At the same time, the bottom is reduced, and the tops are taller and narrower of the peaks.

The parameter of the crystal lattice decreases with the decrease of the temperature, the density increases, the elementary volume of the crystal lattice decreases.

As the temperature decreases, phase transformations take place in special steels by transforming the residual austenite into martensite.

Coefficient of thermal expansion decreases and shows a jump, the density increases, the volume of the elementary cell decreases as temperature decreases. There is no noticeable transformation of white tin into gray tin as the temperature drops.

5. REFERENCES

[1] Bărăian, V.M., *Cercetări asupra proprietăților structurale și fizice ale unor metale și aliaje la temperaturi scăzute,* pp. 79-154, Teză de doctorat, Universitatea Tehnică din Cluj-Napoca, 2004.

[2] Arghir, G., *Caracterizarea cristalografică a metalelor și aliajelor prin difracție cu raze X*, pp. 266-284, Universitatea Tehnică din Cluj-Napoca, 1993.

[3] Bowes, R. G., *The Theory and Practice of sub-zero Treatment of Metals*, pp. 29-32, Heat Treatment of Metals, 1974.

[4] Matei, G., *Teoria structurală a proprietăţii metalelor*, pp. 20-24, vol. I, Institutul Politehnic, Cluj-Napoca, 1986.

[5] Tăpălagă, I., ş.a., *Criogenia în construcţia de maşini,* pp. 178-180, Editura Dacia, Cluj-Napoca, 1988.

STRUCTURA UNOR METALE ȘI ALIAJE LA TEMPERATURI SCĂZUTE

Rezumat: Cercetările experimentale relevă că majoritatea materialelor metalice prezintă, în general, în domeniul temperaturilor scăzute (+15 ... -153 ^oC) reducerea amplitudinii vibraţiei nodurilor reţelei cristaline, fapt reflectat în difractogramele cu raze X; se reduce fondul, iar vârfurile sunt mai înalte şi mai înguste. Parametrul reţelelor cristaline se reduce odată cu scăderea temperaturii, creşte densitatea, scade volumul elementar al reţelelor cristaline. La scăderea temperaturii, au loc transformări de fază în oţelurile speciale prin transformarea austenitei reziduale în martensită. Aceleași fenomene apar la plumb și staniu: scade parametrul cristalografic, coeficientul de dilatare termică prezintă un salt, densitatea crește, volumul celulei elementare scade odată cu scăderea temperaturii. Nu se remarcă transformarea staniului alb în cenușiu.

Vasile-Marius BĂRĂIAN, PhD, Technical University of Cluj-Napoca, Faculty of Materials and Environment, Department of Materials Science and Technology, vm.baraian@gmail.com

George ARGHIR, PhD, Professor Emeritus, Technical University of Cluj-Napoca, Faculty of Materials and Environment, Department of Materials Science and Technology, George.Arghir@stm.utcluj.ro, georgearghir@hotmail.com.