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# Microstructural and Functional Changes Induced by Cryogenic Treatment in 40Cr130, 20Cr130 and X5CrNi18-10 Stainless Steels

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**Abstract**
Cryogenic treatment is an advanced thermal processing technology used to optimise the properties of stainless steels by exposing them to extremely low temperatures. This paper investigates the influence of cryogenic treatment on the microstructure and mechanical behaviour of martensitic 40Cr130 and 20Cr130 stainless steels, as well as on austenitic X5CrNi18-10 steel. The results revealed a strong transformation of the martensitic phases and a significant reduction of residual austenite in the case of martensitic steels, leading to an increase in hardness and, implicitly, wear resistance. In the case of X5CrNi18-10 austenitic steel, cryogenic treatment caused subtle microstructural changes that positively influenced dimensional stability. The study highlights the potential of cryogenic treatment as an effective method for improving the durability and performance of stainless steels, opening new perspectives for its application in industries requiring high reliability and strength.

**Keywords**
steels, heat treatment, cryogenic treatment

## 1. Introduction

Stainless steels are essential materials in numerous industries due to their high corrosion resistance, superior mechanical properties, and durability under extreme environmental conditions [1]. These characteristics make them indispensable in fields such as the chemical, food, medical or aerospace industries. However, to meet the increasingly demanding requirements relating to mechanical performance and wear resistance, there are sought effective methods to improve the properties of these alloys [4].

Cryogenic treatment is an innovative technology of subjecting materials to extremely low temperatures (down to –196 °C or below) in order to modify the internal structure and microstructure of metals [2, 3]. This process can induce phase transformations, residual stress reduction, and a more uniform distribution of carbides, leading to increased hardness, wear resistance, and dimensional stability [5].

Although cryogenic treatment is well studied for tool steels and other ferritic-martensitic materials, its application to stainless steels, especially austenitic and martensitic, is a relatively new field, which promises significant improvements, but whose mechanism is not yet clearly understood. This paper aims to investigate the influence of cryogenic treatment on the resilience of the studied stainless steels, thus providing insight into the potential and limitations of this method.

## 2. Experimental Research

In industrial practice, as in everyday life, many of the components that make up devices and aggregates may undergo significant, single or repeated cooling. If temperatures drop significantly, they can reach values below the Mf points of the alloys used, causing phase transformations. Phase transformations in combination with the stiffening process, induced by low temperatures, can adversely impact the mechanical characteristics of the material and, therefore, of the assembly or subassemblies on which a certain component operates. Most of the time, temperatures significantly decrease the impact strength of the given material. This effect can manifest itself both at negative temperatures and after heating to ambient temperature.

Impact strength is affected both in materials with phase transformations and in those in which such processes do not occur.

Temperature was measured using thermocouples. The final calculation of the fracture energy includes a correction factor imposed by the occurrence of concentrators in the body of the specimen, as well as the fracture energy of the plastic sheath (previously determined).

Three stainless steel grades were used in the research, and they are presented in Table 1.

Table 1. Materials used in the study

| Steel | Structural type | Main content (%) | Typical applications |
|---|---|---|---|
| 20Cr130 | Martensitic | ~13% Cr, ~0.2% C | Valves, springs, tools |
| 40Cr130 | Martensitic | ~13% Cr, ~0.4% C | Tools, bearings, wear-resistant components |
| X5CrNi18-10 | Austenitic | 18% Cr, 10% Ni | Food, chemical equipment, tanks |

The specimens used in the tests were 50×10×10 mm, with a V-shaped notch.

The tests were carried out under the following conditions:

- cooling to −80 °C in an enclosure with automatic temperature control with an accuracy of +/− 1 °C;
- cooling to −196 °C by immersion in liquid nitrogen at −196 °C;
- the resilience test at −80 °C was performed by the rapid transfer of the specimen to the fracture grips, and they were fractured at −80 °C (+/− 1°C);
- the resilience test at 20 °C was performed with the specimen installed on the grips, the temperature was measured up to 20 °C (+/− 1°C), when the fracture was performed.

The tables below present the cryogenic tests performed on specimens made of the aforementioned steels, as well as the resilience values obtained. For each case, three samples were tested.

Specimens made of 40Cr130 steel were heat treated according to the data in Table 2.

Table 2. Cryogenic treatments applied to 40Cr130 specimens

| Specimen | Heat treatments | | Cryogenic treatments | | | | Fracture energy KCU [J/cm²] |
|---|---|---|---|---|---|---|---|
| | Quenching in vacuum [°C] | Tempering in vacuum [°C] | Cooling temperature [°C] | Holding time [min] | Cooling medium | Fracture temperature [°C] | |
| 1 | 1040 | 510 | - | - | - | Ambient temperature (control sample) | 8.7 |
| 2 | | | −(85-90) | 30 | Liquid nitrogen | −80 | 7.6 |
| | | | | | | 20 | 8.3 |
| 3 | | | −196 | 5 | Liquid nitrogen | −80 | 5 |
| | | | | | | 20 | 5.8 |
| 4 | | | −196 | 30 | Liquid nitrogen | −80 | 3.4 |
| | | | | | | 20 | 3.7 |

Compared to the control sample, all cryogenically treated samples showed lower, sometimes spectacular, resilience values.

The decrease in cryogenic temperature from −85.... −90 °C to the liquid nitrogen temperature of −196 °C caused a further decrease in resilience values.

Reheating the samples to ambient temperature, after deep cooling, resulted in a slight increase in resilience, compared to samples tested at −80 °C.

The decrease in resilience values due to undercooling can have the following causes: phase transformations, material stiffening, cold working caused by the shrinkage of the metallic material due to the drastic decrease in temperatures.

By cooling to −196 °C, it is possible to completely transform residual martensite into martensite, with an appropriate decrease in impact strength.

The accuracy of measuring temperatures and holding time during the test was +/− 1 °C; 5 sec.

All the specimens tested from negative to ambient temperatures suffered brittle failure - in the fracture cross-section, the samples had a crystalline, uniform, very fine appearance. Figures 1… 5 show the appearance of specimens fractured under cryogenic conditions and their structures.

Fig. 1. Specimen three fractured (breaking section) at –80 °C after immersion in liquid nitrogen for five minutes, down to –196°C

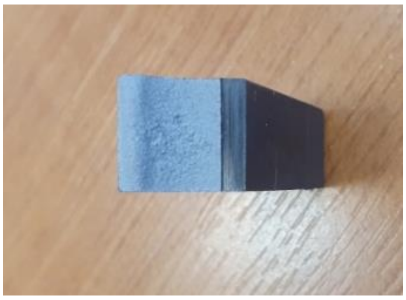

Fig. 2. Specimen four fractured (breaking section) at –80 °C immersed in liquid nitrogen, for 30 minutes, down to –196 °C

Fig. 3. 40Cr130 hardened at 1040 °C, tempered at 510 °C held in liquid nitrogen vapours for 30 minutes at – (85-90) °C and fractured at –80 °C. Etching reagent: aqua regia. 100:1

Fig. 4. 40Cr130 hardened at 1040 °C, tempered at 510 °C held in liquid nitrogen vapours for 30 minutes at – (85-90) °C and fractured at –80 °C. Structure: martensite + carbides. Etching reagent: aqua regia. 500:1

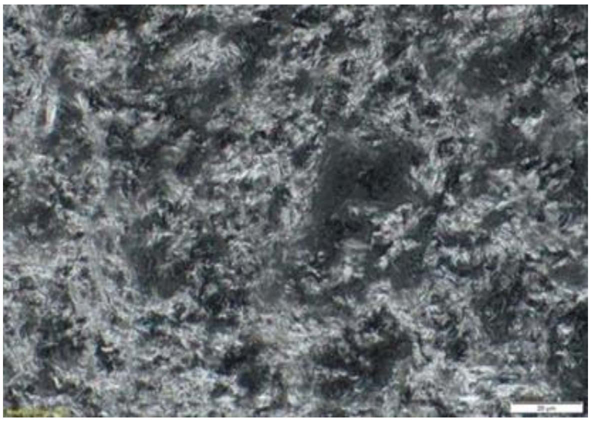

Fig. 5. 40Cr130 hardened at 1040 °C, tempered at 510 °C held in liquid nitrogen vapours for five minutes at –196 °C (liquid nitrogen temperature) and fractured at –80 °C. Fracture core structure. Etching reagent: aqua regia. 1000:1

A similar procedure was followed for the other materials. Tables 3 and 4 present the resilience results obtained after various treatments.

Table 3. Fracture energy following heat and cryogenic treatments applied to 20Cr130

| Heat treatment | | Cryogenic treatment | | | | Resilience KCV [J/cm²] |
|---|---|---|---|---|---|---|
| Vacuum quenching [°C] | Vacuum tempering [°C] | Cooling temperature [°C] | Holding temperature [°C] | Cooling medium [°C] | Fracture temperature [°C] | |
| 1040 | 510 | - | - | - | - | 26 |
| | | − (85-90) | 30 | Liquid nitrogen | −80 | 6.2 |
| | | − (85-90) | 30 | Liquid nitrogen | 20 | 22 |
| | | −196 | 5 | Liquid nitrogen | −80 | 6 |
| | | −196 | 5 | Liquid nitrogen | 20 | 23 |

Table 4. Fracture energy following heat and cryogenic treatments applied to X5CrNi18-10

| Heat treatment | | Cryogenic treatment | | | | Resilience KCV [J/cm²] |
|---|---|---|---|---|---|---|
| Vacuum quenching [°C] | Vacuum tempering [°C] | Cooling temperature [°C] | Holding temperature [°C] | Cooling medium [°C] | Fracture temperature [°C] | |
| 1040 | 510 | - | - | - | - | Not fractured |
| | | − (85-90) | 30 | Liquid nitrogen | −80 | 32 |
| | | − (85-90) | 30 | Liquid nitrogen | 20 | 40 |
| | | −196 | 5 | Liquid nitrogen | −80 | 32 |
| | | −196 | 5 | Liquid nitrogen | 20 | 41 |

It follows from the above that these two steels are also sensitive to deep undercooling in terms of resilience. The rupture at −80 °C had the lowest impact strength values. By reheating to ambient temperature, the resilience values increase, but the values of the control samples that underwent no undercooling cannot be reached. This indicates that changes have occurred in the structure, without being essential, and the final values are slightly lower than the reference values in the case of 20Cr130.

In the case of austenitic steel, resilience through cryogenic treatment clearly decreases compared to the control samples in which toughness could not be determined numerically, as only plastic deformation occurred, no fracture. Cooling to −80 °C and −196 °C led to a decrease in toughness, which also confirms the existence of structural transformations based on the cold working of the material due to thermal shrinkage; however, phase transformations may exist in small quantities in the volume of the material.

The comparative study on the influence of deep undercooling of the three steels on impact strength is summarised in Table 5.

Table 5. Comparative study on the results of cryogenic treatment

| Cryogenic treatment | | Resilience KCV [J/cm²] | | |
|---|---|---|---|---|
| Cooling temperature [°C] | Fracture temperature [°C] | 40Cr130 | 20Cr130 | X5CrNi1810 |
| - | - | 8.7 | 26 | Not fractured |
| −(85-90) | −80 | 8.3 | 6.2 | 32 |
| | 20 | 7.6 | 22 | 40 |
| −196 | −80 | 5 | 6 | 32 |
| | 20 | 5.8 | 23 | 41 |

Table 5 illustrates that, in all the cases, the cooling of the given steels influences their impact behaviour, i.e. toughness decrease. The data obtained shows that cooling to −80 °C influences to the greatest extent in terms of reducing resilience; as a result, in the 20 °C ... −80 °C range occur the most important phenomena that cause embrittlement. These may be the formation of microcracks in the material hardened by cooling and minor phase transformations to a lesser extent.

At the negative temperatures at which some tests were performed, the cold stiffening of the metallic material has a major influence. This stiffening partially disappears upon returning to ambient temperature.

## 3. Conclusions

Cryogenic treatment applied to 40Cr130 and 20Cr130 martensitic stainless steels demonstrated a significant impact on their microstructure and mechanical properties. The transformation of residual austenite into martensite and the fine precipitation of carbides led to increased hardness and wear resistance, thus improving the performance of these alloys under intense mechanical stress. Moreover, the reduction of internal stresses contributes to better dimensional stability of the treated components.

In the case of X5CrNi18-10 austenitic stainless steel, the cryogenic treatment generated subtle microstructural changes, which influenced dimensional stability, without compromising the characteristic ductility of this class of steels. These results indicate that cryogenic treatment can also be successfully applied to austenitic stainless steels, expanding the area of use of this technology.

Overall, the study highlights the potential of cryogenic treatment to improve the durability and mechanical performance of stainless steels used in industries with high reliability requirements. The implementation of this treatment can lead to increased component lifespan and reduced maintenance costs, thus recommending its integration into industrial technological processes.

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