Abstract

Steels parts used in helicopters are critical in terms of mechanical performance and safety issues where failure can lead to catastrophic consequences. The objective of this study was to evaluate the influence of low temperatures on toughness, through impact testing, on 45NiCrMo16 steel alloy used in the manufacture of bolts used on a helicopter rotor. The aircraft that the studied bolt is used can operate in the Antarctic environment. The temperatures used in the Charpy tests were -40 ºC, -20 ºC, 0 ºC, and 25 ºC. Besides, microstructural analysis (optical and scanning microscopy) and microhardness were done to identify the steel phases/microconstituents and any surface treatment presence. The results showed the presence of a hardened layer, a decrease in impact toughness with decreasing temperature, and a transition from ductile to brittle fracture. The conclusions indicate behavior that can restrict or limit this steel grade application in extreme temperature conditions, that is the ductile-brittle transition region begins from a decrease in temperature starting from -20 ºC.

Keywords: Bolt; Temperature; Toughness; Ductile-brittle transition.

1 Introduction

Ever since the beginning of the 19th century, extremely low temperatures were discovered as the cause of the toughness decrease of metallic material, which has encouraged research to determine the characteristics and behavior of the metallic materials at low temperatures [1]. Inefficient manufacturing practices, combined with low material strength, as well as inadequate design specifications, contributed to these failures. The history of fragile fracture includes 160 ships, which had catastrophic failures during the winter months between 1943 and 1944 due presence of low temperatures, erroneous design (stress concentration) and weld defects and discontinuities. Failures in storage tank also occurred on different occasions in 1919 and 1978 [2]. Research for the development of high strength steel alloys for low-temperature applications that can withstand critical loads in extreme environments have been done [3-6].

Yan et al. [3] evaluated and compared tensile properties of mild steel and high-strength S690 steel plates in low temperature. Based on their test results, for the high strength S690 steel plates, the average elastic modulus, yield strength, tensile strength and fracture strain values increased by 13%, 7%, 9%, and 4.5%, respectively when the temperature decreases from 30 ºC to -80 ºC. They pointed that the influence of the low temperature on these mechanical properties of the high strength steel was less significant compared to those of the mild steel plates with the same thickness. Mendagaliyev et al. [6] reported results of direct laser deposition (DLD) of cold-resistant steels. They processed 09CrNi2MoCu steel powder by DLD and found tensile strength and elongation properties higher than in rolled products due to volumetric thermal cycling occurring in the process of plowing. They point out that DLD process is a promising technology that is not inferior to traditional methods of production reducing the intermediate process cycles, and subsequent heat treatment.

The operation of aircraft and usage of structures in hostile environments of low temperatures should be planned and executed with a high level of detail, given the extreme weather conditions, cold weather, and difficulty in logistical support and supplies [7]. Bolts are defined as aircraft safety parts since they effectively carry part of the weight of the helicopter during the flight. Given its role in the structural integrity of the helicopter, the failures of the structure bolts can have severe or fatal consequences for the safety of the crew and passengers and the aircraft itself [8-10]. Other mechanical components are safety-critical parts as well and have been researched objects, e.g., transmission shaft, crankshaft and main rotor grip [11-13].

Krstic et al. [8] performed a failure analysis investigation on the mounting bolt of helicopter main gearbox support strut.
They reported that due to significant damage of the cadmium protective layer corrosion products within the all engaged threads of the bolt-nut interface are present, that it caused cracks that originated in the thread roots and propagated under tensile load. That Mi-8 helicopter main gearbox rear left bracket mounting bolt failed due to hydrogen assisted stress corrosion crack. Lee et al. [10] conducted research on the failure analysis of the collapsed clamp bolt from a helicopter engine. Through fractography, metallography, and stress analysis of the failed part, it was found that the clamp bolt was fractured by stress corrosion cracking due to the interaction of tensile residual stress and corrosive environment.

In this context, it is necessary to establish data from the ductile-brittle transition of metallic materials used in helicopters designed to operate at cryogenic temperatures. Therefore, this study aims to characterize the steel used in a helicopter rotor bolt to provide more information for engineers, designers, and aircraft users. Such data are fundamental for failure analysis and mainly to prevent accidents from occurring.

2 Materials and methods

The analyzed component is a bolt made of steel that is used in the main rotor of an aircraft (Figure 1a) of rotating wings and has, in its fixation part, the bolt under analysis (Figure 1b).

The bolt was analyzed after the preventive maintenance of the aircraft and was removed from the aircraft because it reached the number of flight hours defined by the manufacturer, although it did not present wear or malfunction. The helicopter manufacturer’s policy adopts the non-providing of material details such as chemical composition or heat/thermochemical treatment of the bolt employed. The dimensions of the bolt were 220 mm long and 24 mm in body diameter.

For microstructural analysis, a cross-section of the bolt body located just after the bolt head was removed using an abrasive cutting disc with lubrication. The samples were prepared with silicon carbide sandpaper until #1200 mesh sandpaper and subsequent polishing with a felt disc with abrasive alumina solution, and 3% Nital etching (3% nitric acid and 97% alcohol) was used to reveal the microstructure.

The equipment used to determine the chemical composition of the bolt base metal was Spectro Lab from SPECTRO through optical emission spectroscopy. The optical microscopy it was performed using Olympus GX 51S. The examination of the surface treatment and precipitates by scanning electron microscope (SEM) was done utilizing JSM - 6610LV from JEOL.

A microhardness test was performed to measure the hardness profile of the material according to ASTM E384 [14]. The distance between indentations of 0.15 mm was used with a load of 300 g and a loading time of 12 s. The measurements were made from the surface to the core, with the first indentation being made approximately 0.4 mm away from the edge of the sample.

The preparation of the specimens for the Charpy impact test was performed using the bolt milling and finishing with grinding by ASTM E23 [15]. In total, twelve samples were tested, three samples at each temperature, under the conditions of -40 ºC, -20 ºC, 0 ºC, and 25 ºC with type C notch (Charpy-U). The samples were cooled through a sealed temperature stabilization tank (Figure 2) and temperature measurement using K thermocouple. Sub-zero temperatures were reached with the use of liquid nitrogen.

3 Results and discussions

The chemical analysis results presented in Table 1 showed that the material is similar to the steel grade 45NiCrMo16 and exhibited medium carbon content and nickel, chrome, and molybdenum as alloy elements. The addition of Ni, Cr and Mo brings a balance between strength, toughness, and hardenability, being suitable for thermochemical heat treatment [14]. Other researchers have reported studies done on bolts used in helicopters with metal alloys different than the present study. Krstic et al. described a failure of mounting chromium-nickel steel bolt of helicopter main gearbox support strut, they significant damage of the cadmium protective layer and hydrogen assisted stress corrosion cracking [8]. Eliaz et al. discussed failures of Inconel 718 bolts in helicopter

![Figure 1](image1.png)

**Figure 1.** (a) Main rotor of an aircraft (b) evaluated fixing bolt, scale bar length equals to 50 mm.
Due these different features noted between the surface and the core observed on the Figure 4, dispersive energy spectroscopy (EDS) was performed by scanning electron microscopy (SEM). This EDS results demonstrated different chemical concentration between the surface and the core.

Table 2 shows EDS analysis results from the spots starting from the surface region towards the bolt core, these spots are indicted within the Figure 5. Even though EDS analysis is not precise for carbon, a higher amount of carbon was detected suggesting thermochemical treatment existence, moreover the presence of nitrogen at significant concentrations was identified. X-ray diffraction (XRD) analyses as well were performed, but the results were not conclusive for reliable identification of carbonitrides or other compounds. The low and undetectable amount of secondary carbides and nitrides obtained on the performed XRD analyses may be associated with an insufficient initial base material amount of Nb, V, Ti and Al on the bolt base material associated with thermochemical treatment parameters applied on the bolt manufacture process [16-18]. Krstic (8) as well detected chromium carbides precipitates on the surface of bolts made of chromium-nickel steel used on helicopters.

Figure 6 shows the microhardness profile along the cross-section of the bolt. The maximum measured value was 800 HV, and the core value was 450 HV. The maximum value within the layer and thickness size observed is compatible with the carbonitriding process, according to Totten [16]. Also, the microstructure observed on the surface and the core and mainly the chemical composition values obtained corroborate and well-matched with typical carbonitriding product characteristics [19]. Carbonitriding is an efficient thermochemical heat treatment used to improve surface properties, as it increases hardness and, consequently, wear
resistance. Besides contributing beneficially to fatigue resistance due to the residual stress profile provided by the process [20].

The hardened layer presented in the bolt was not considered for the Charpy tests, as it was removed given the need to machining the samples to meet the size specifications by the ASTM E23 [15].

The values obtained through the Charpy tests (Figure 7) were plotted in as a curve (Figure 8) to establish the ductile-brittle transition of the steel analyzed. Each temperature condition was tested using three samples in each, i.e., at temperatures of -40 °C, -20 °C, 0 °C, and 25 °C. It was observed that the mean values obtained at -20, 0, and 25°C showed small differences reaching a plateau of absorbed energy, nonetheless the standard deviation for 0°C was more significant. However, in the test condition at -40°C of temperature, a considerable decrease in absorbed energy was observed. The toughness decrease with temperature decrease is already expected because it is a characteristic of materials with a crystalline structure of body-centered cubic

Table 2. Result of the analysis done by EDS

<table>
<thead>
<tr>
<th>wt %</th>
<th>C</th>
<th>N</th>
<th>Si</th>
<th>Cr</th>
<th>Fe</th>
<th>Ni</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spot 1</td>
<td>1.21</td>
<td>1.78</td>
<td>0.56</td>
<td>2.11</td>
<td>90.13</td>
<td>3.77</td>
<td>0.43</td>
</tr>
<tr>
<td>Spot 2</td>
<td>1.06</td>
<td>1.94</td>
<td>0.63</td>
<td>1.90</td>
<td>89.78</td>
<td>4.00</td>
<td>0.69</td>
</tr>
<tr>
<td>Spot 3</td>
<td>1.30</td>
<td>1.99</td>
<td>0.56</td>
<td>2.21</td>
<td>90.17</td>
<td>3.77</td>
<td>0.65</td>
</tr>
<tr>
<td>Spot 4</td>
<td>1.04</td>
<td>1.72</td>
<td>0.38</td>
<td>2.19</td>
<td>90.78</td>
<td>3.52</td>
<td>0.37</td>
</tr>
<tr>
<td>Spot 5</td>
<td>1.00</td>
<td>1.64</td>
<td>0.55</td>
<td>2.31</td>
<td>90.10</td>
<td>3.69</td>
<td>0.71</td>
</tr>
</tbody>
</table>

Figure 4. Bolt surface indicating presence of precipitates (indicated by arrows).

Figure 5. Measuring spots analyzed by EDS.

Figure 6. Bolt microhardness profile

Figure 7. Absorbed energy as a function of the temperature for the Charpy tests.

Figure 8. The ductile-brittle transition curve obtained by the Charpy tests.
(BCC), presenting this decrease in toughness with the drop of the temperature. However, the presence of precipitates in the material matrix plays an essential role in the initiation of cleavage fracture [21], which may thus have contributed to the significant decrease in energy absorbed at -40 °C tested temperature. An effective approach to increase the toughness of steels is to increase the alloy’s cleanliness, especially in the decrease of sulfur and phosphorus contents [2].

Kantora and his colleagues enlightened the connection between occurrence of remarkable impact toughness scattering in ductile-brittle transition region and microstructure features of low carbon microalloyed steels. The formation of pancaked parent austenite microstructure, various ferritic microstructures and martensite-austenite constituents along with significant grain size variations could be considered as the sources for the occurrence of remarkable impact toughness scattering [22]. Nevertheless, the entire behavior of the ductile-brittle transition could be accurately determined performing more tests at temperatures below -40 °C to establish the lower level of the transition curve. According to the tests done, the ductile-brittle transition region begins from the decrease in temperature starting from -20 °C.

4 Conclusions
The steel used in the manufacture of the bolt had a chemical composition equivalent to the grade 45NiCrMo16. Presence of surface treatment consistent with a carbonitriding with a hardened layer of 0.352 mm depth, maximum hardness of 850 HV and a martensitic core microstructure. The Charpy tests at -20, 0, and 25 °C showed similar values with absorbed energy around 69 J and a significant drop at -40 °C of 45 J.

The ductile-brittle transition region begins from a decrease in temperature starting from -20 °C.

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