

Industrial scale extrusion performance of cryogenically processed DIN 100 Cr6 and DIN 21NiCrMo2 steels

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ABSTRACT: The effects of different heat and cryogenic (sub-zero) treatment parameters such as temperature and holding time on the microstructure (amount of retained austenite) and hardness of extrusion molds produced from the 21NiCrMo2 and 100Cr6 steels were investigated. The 21NiCrMo2 grade extrusion die was carburized for 22.5 h in an endogas (25% CO, 35% N₂, 40% H₂) atmosphere at 920 °C. At the end of the carburization process, the temperature was kept at 850 °C, which is the austenitization temperature, for 2 h, followed by cooling in oil at 80 °C and remaining in oil for 45 minutes. The carburizing process was not performed for the extrusion molds made of 100Cr6 steel grade. Only the austenitizing heat treatment at 850 °C (holding for 2 h) was carried out in this steel. The steel molds which were produced with 21NiCrMo2 and 100Cr6 steels were cryogenically treated at -120 °C for 2 h and subsequently tempered at 150 °C for 1.5 h. As a result of the cryogenic treatment, the hardness of 21NiCrMo2 steel increased to 840 Hv and the wear resistance of the extrusion die surface was improved. The amount of residual austenite decreased from 20% to 6% after the cryogenic treatment. Due to the effect of the cryogenic process, the surface hardness of the 100Cr6 steel sample increased to ~870 Hv, which implies an increase of 4.5%, due to the transformation of residual austenite to martensite. The mass loss, during the wear tests, of the hardened extrusion dies was reduced from 0.1420 mg to 0.0221 mg. The notch impact strength value measured in this condition was 20 J. The 100Cr6 steel after the cryogenic treatment was used to extrude 12 tons of Al alloy in an industrial press. This amount of material is 30% lower than for hot work tool steel. On the other hand, the 100Cr6 steel is more economical and heat treatment is more practical. The extrusion performance of 21NiCrMo2 steel was 50% lower than the hot work tool steel.

KEYWORDS: Cryogenic treatment; Extrusion Die; Hardness; Retained austenite; Steel; Wear

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RESUMEN: Resistencia al desgaste y rendimiento durante procesos de extrusión a escala industrial de los aceros 100Cr6 y 21NiCrMo2 sometidos a tratamientos criogénicos. Se ha investigado los efectos de diferentes parámetros de tratamiento térmico y criogénico como la temperatura y el tiempo de mantenimiento sobre la microestructura

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(cantidad de austenita retenida) y la dureza de moldes de extrusión producidos a partir de los aceros 21NiCrMo2 y 100Cr6. La matriz de extrusión del acero 21NiCrMo2 se cementó durante 22,5 h en una atmósfera de gas (25% CO, 35% N₂, 40% H₂) a 920 °C. Al final del proceso de cementación, la temperatura se mantuvo a 850 °C, que es la temperatura de austenización, durante 2 h, seguido de enfriamiento en aceite a 80 °C, permaneciendo en aceite durante 45 minutos. No se realizó este proceso de cementación para los moldes de extrusión fabricados con el acero 100Cr6. En este acero solo se llevó a cabo el tratamiento de austenización a la temperatura de 850 °C (manteniendo durante 2 h). Los moldes de acero que se fabricaron con aceros los 21NiCrMo2 y 100Cr6 se trataron posteriormente de manera criogénica a -120 °C durante 2 h y, posteriormente, se templaron a 150 °C durante 1,5 h. Como resultado del tratamiento criogénico, la dureza del acero 21NiCrMo2 aumentó hasta los 840 Hv y mejoró la resistencia al desgaste de la superficie de la matriz de extrusión. La cantidad de austenita residual disminuyó del 20% al 6% después del tratamiento criogénico. Por efecto del proceso criogénico, la dureza superficial de la muestra de acero 100Cr6 aumentó a ~870 Hv, lo que supone un incremento del 4,5%, debido a la transformación de la austenita residual a martensita. La pérdida de masa durante el ensayo de desgaste de las matrices de extrusión endurecidas se redujo de 0,1420 mg a 0,0221 mg. El valor de resistencia al impacto medido en esta condición fue de 20 J. El acero 100Cr6 después del tratamiento criogénico se usó para extruir 12 toneladas de aleación de Al en una prensa industrial. Esta cantidad de material es un 30% inferior a la del acero para herramientas para trabajo en caliente. Por otro lado, el acero 100Cr6 es más económico y el tratamiento térmico es más práctico. El rendimiento durante el proceso de extrusión del acero 21NiCrMo2 fue un 50% inferior al del acero para herramientas de trabajo en caliente.

PALABRAS CLAVE: Acero; Austenita retenida; Desgaste; Dureza; Moldes de extrusión; Tratamiento criogénico

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1. INTRODUCTION

The application of cryogenic treatments to metals has recently been recognized as an effective method to increase the “wear resistance” and reduce residual stresses in tool or die steels (Paulin, 1993; Das *et al.*, 2009; Yan and Li, 2013; Mazor *et al.*, 2017; Ptačinová *et al.*, 2017; Zhang *et al.*, 2019) and, unlike surface modifications and coatings, it is an economical, one-time permanent process that affects the interior phase structure of steels. A cryogenic treatment is an additional process to conventional heat treatments a is used in steel samples that contain retained austenite to promote its transformation to martensite. In this process, the samples are cooled below room temperature, held at this temperature until a microstructural phase transformations takes place, and then heated back to room or tempering temperature (Pillai *et al.*, 1986; Das *et al.*, 2010). In applications with a cooling temperature in the range of -125 to -196 °C, defined as deep cryogenic processes, certain material properties have been improved beyond the results achieved by the cooling process at higher temperatures below room temperature (Collins and Dormer, 1997; Yen and Kamody, 1997; Kalsi *et al.*, 2010; Mazor *et al.*, 2017). Certain materials properties result from the formation of very small carbides dispersed in the tempered martensitic structure as well as the complete transformation from austenite to martensite (Paydar *et al.*, 2014; Villa *et al.*, 2014; Jurčí *et al.*, 2017). Cryogenic treatments are an extra mechanical property enhancement processes applied between quenching to room temperature and tempering processes. It has been re-

ported that further improvement can be obtained by performing the process at the end of the usual heat treatment cycle, for example surface grinding in finished processes, (Popandopulo and Zhukova, 1980; Slatter and Thornton, 2017). On the other hand, the wear properties, toughness, phase and microstructure characterization of 21NiCrMo2 steel, which surface is carburized using endogas for their use in extrusion dies, are not at a sufficient level in the literature. In this study, carburized 21NiCrMo2 steel and heat treated 100Cr6 steel were subjected to cryogenic treatments and their toughness and wear resistance tested. As a case study, billet production was made in the pilot scale extrusion dies and the results were compared.

2. MATERIALS AND METHODS

2.1. Materials

The process flow diagram is organized in the form of turning, milling and grinding, respectively. The steel was first turned into a lathe by cutting the front/back sides of the mill and the milling process was completed with the threading process. Finally, the production of the mold is completed by grinding of the outer corner and the grinding of the bearings. The chemical composition of the steels used to produce the extrusion molds is shown in Table 1.

2.2. Heat and cryogenic treatments

Extrusion mold produced using the two steels under investigation were grinded and different heat treatment cycles have been applied. 21NiCrMo2

TABLE 1. Chemical composition of 21NiCrMo2 and 100Cr6 steels (in wt.% with Fe to balance).

	C	Mn	Ni	Cr	Mo	Si	S	P	Cu
21NiCrMo2	0.21	0.77	0.43	0.55	0.18	0.20	0.02	0.026	-
100Cr6	0.91	0.33	-	0.47	0.06	0.27	0.01	0.023	0.29

samples grade extrusion molds were carburized at 920 °C in endogas (25% CO, 35% N₂, 40% H₂) atmosphere for 22.5 h. The extrusion molds made of 100Cr6 was not subjected to the carburizing because it has enough carbon content. 100Cr6 extrusion molds were heated to the austenitizing temperature of 850 °C and held for 2 h. Then all samples (21NiCrMo2, 100Cr6 extrusion molds) were hardened in oil at 80 °C by promoting the formation of martensite after cooling to this temperature (and held for 45 min). After the hardening process, samples were cryogenically treated by using liquid Nitrogen at -120 °C for 2 h. Then samples were tempered at 150 °C for 2.5 h to minimize the residual stresses in the extrusion mold. The shallow cryogenic treatment which undertaken between -80 °C and -130 °C was performed. The shallow cryogenic treatment is defined as the most proper process for this alloy. Table 2 shows the heat treatments applied to the extrusion molds of different materials.

2.3. Metallographic studies and microscopy observations

All extrusion die steels, which were subjected to cryogenic treatments and then tempered, were made ready for microstructure examination by standard metallographic methods and then the samples were etched using 2% Nital solution. Afterwards, examinations were carried out with optical and scanning electron microscopes. The Tescan Vega test device was used.

2.4 Mechanical tests

2.4.1. Hardness test

The hardness values were measured on the cross-sectional area from the extrusion molds. Mi-

cro hardness test were performed using a 1 kgf load. Hardness measurements were tested from the surface to the center of the sample. Hardness measurement values are obtained separately for each heat treatment.

2.4.2. Wear test

For the wear tests, wear test samples were undertaken on the cross-sectional area on the extrusion mold under untreated and heat treated conditions. Pin-on disc type of test apparatus (Koehler Instrument, K93590 Pin on Disc Tester 230V, 50 Hz) was used for the wear tests. These tests were conducted in accordance with the ASTM G99-05 (2010), with various loads, constant distance and at a constant RPM. Then, weight loss was measured by a sensitive balance and then the wear surfaces of each sample were examined. Because the ASTM G99-05 (2010) standard is based on the measurement of the wear volume, the wear on the ball bearing was calculated by using a mathematical relationship and direct measurements of the volumetric loss. Wear tests were performed at room temperature with samples of 5 mm in diameter, under a load of 30 N, a rotation speed of 300 rpm, along a distance of 500 m.

2.4.3. Impact test

Notch impact test were conducted on a Izod Charpy test device with a capacity of 350 J under different conditions. Test specimens were adjusted to the dimensions: 55×10×10 mm³ as a standard. The experiments performed on the treated samples were conducted at an ambient temperature of 18 °C and in 45% humid air atmosphere and each test was repeated twice. As a result of the impact tests, the refractive surface morphology was examined by performing SEM analyses.

TABLE 2. Processing steps applied to the steels under investigation.

Material	Carburized	Austenitized	Oil Quench	Cryogenic	Tempered
21NiCrMo2	+	+	+	+	+
100 Cr6	-	+	+	+	+

2.5. Extrusion test

The performance of the extrusion molds after applying the heat and cryogenic treatments under factory operating conditions was examined. For these experiments, extrusion die life and billet production capacities obtained under routine production conditions were compared with cryogenic process die extrusion performances. Routine casting and extrusion parameters were used in the production of 6000 series Al alloys in the company. First of all, the homogenisation process of the 6060 aluminium billets was carried out and then the extrusion process was performed for the aluminium profile production. The mold without heat treatment was connected to the extruder and extruded with an extrusion speed of 5 mm·s⁻¹ by applying 30 and 75 extrusion rates (R). The extrusion speed was controlled with the aid of the punch advance-time curve. The billet and shell temperature, which are defined as process parameters, were kept constant at 450 °C and 400 °C respectively.

3. RESULTS

Figure 1 shows the microstructure of the samples after 2%-Nital etching. After the heat treatment, the microstructural studies revealed that martensite phase was formed on the steel surface. The martensitic phase transformation occurs by diffusion-free phase transformation. Martensitic transformation is a diffusionless, military-type of phase transformation in which atoms move with slip-like mechanisms (Otsuka and Wayman, 1998).

The microstructure of these alloys after the heat and cryogenic treatments contained retained austen-

ite and martensite phase which is a metastable low temperature phase (Ivanić *et al.*, 2014). Austenite is stable at high temperature but might be retained at room temperature depending on the alloy composition and applied heat treatment. When the temperature is increased, the decomposition of martensite is promoted. If the phase transformation occurs, the martensite turns into the main façade in the same direction from the other (Funakubo, 1987). In the measurements undertaken on the surface of the samples, a residual austenite of 15-25% was encountered. According to the cryogenic treatment results, the amount of retained austenite obtained in the DIN 100Cr6 steel is higher than that of 21NiCrMo2.

4. DISCUSSION

The plots in Fig. 2 show the hardness measurement results from the surface of the specimen up to a depth of 2 mm for the two steels investigated in this research. The carburizing process was only performed for 21NiCrMo2 for 22.5 h in an endogas atmosphere at 920 °C. The austenitizing temperature for 21NiCrMo2 and 100Cr6 steels was set to 850 °C and held for 2 h. Also these steels were cryogenically treated at -120 °C for 2 h and subsequently tempered at 150 °C for 1.5 h. After the carburizing process the surface hardness increased to 800 Hv. After the cryogenic treatment, the surface hardness of the 100Cr6 sample has increased to ~850 Hv. According to the results of the hardness measurements, the hardness of the cryogenic treated parts increased by 4.5%. Cryogenic treated specimens were tempered and, as a result, 6% reduction in hardness was measured. The 21NiCrMo2 and 100Cr6 steels showed a much pronounced

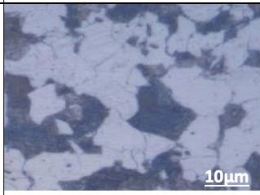
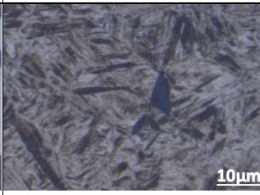
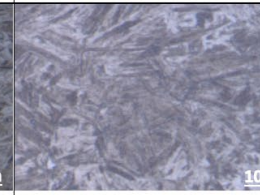
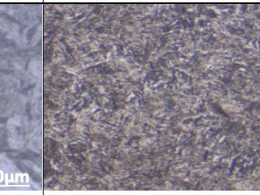
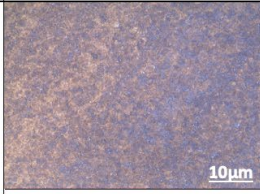
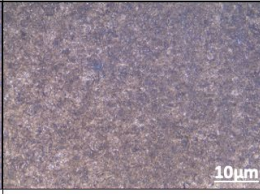
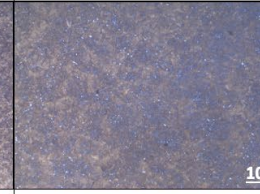
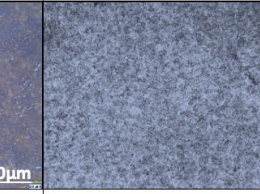
	Untreated	Hardened	Cryogenic	Tempered
21NiCrMo2				
RA	48 %	21 %	6 %	5 %
100Cr6				
RA	53 %	23 %	7 %	6 %

FIGURE 1. Light optical micrographs of the steel microstructures before and after the different heat and cryogenic treatments (Symbol RA stands for retained austenite).

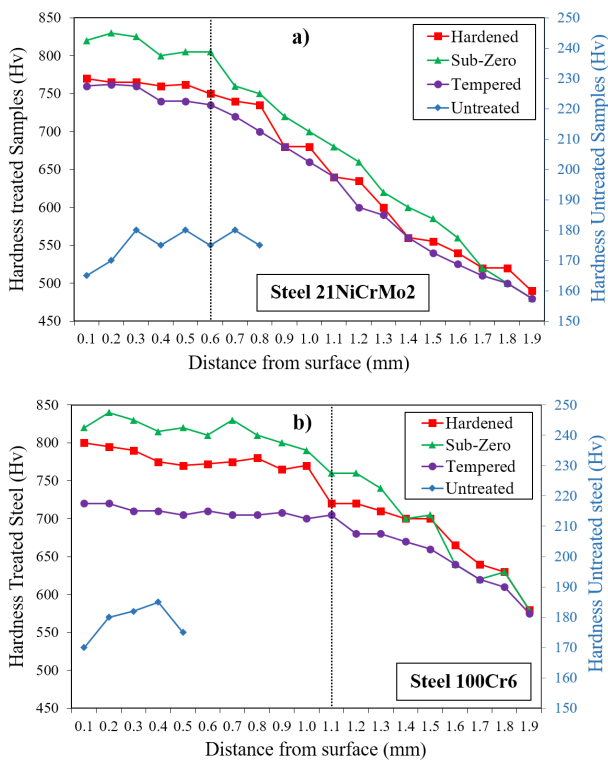


FIGURE 2. Hardnesses profile measured after the different heat and cryogenic treatments in steels: a) 21NiCrMo2; b) 100Cr6.

decrease in hardness after a depth of ~0.6 and 1.0 mm, respectively; up to this depth distance, the hardness remains roughly stable in the tempered condition and then drops. The reason for the high hardness of the

100Cr6 steel is due to the Manganese content. The effect of the applied cryogenic process on hardness is decreased after a depth of ~1.0 mm. Figure 1 clearly shows the transformation from retained austenite to martensitic in the microstructure. The amount of retained austenite decreased from 48% to 6% after the cryogenic treatment for steel 21NiCrMo2. For steel 100Cr6 the amount decreased from 53% to 7% after the cryogenic treatment.

The friction coefficient-wear distance graphs of the untreated and hardened samples against Al₂O₃ balls are given in Table 3. After testing 500 m distance under 30 N load, the mean friction coefficients measured for the untreated samples were 0.5864 and 0.6321, respectively, while lower friction coefficients were observed in the cryogenic samples (Table 3). The reason for the high coefficient of friction is that it causes abrasive wear. The surface hardness of the samples increased after the cryogenic treatment and the friction coefficient decreased with increasing surface hardness for each steel. The lowest friction coefficient after the whole heating/cryogenic cycle was measured in steel 100Cr6 (determined to be 0.2789) with a weight loss of 0.0221 mg, after testing 500 m distance. The experimental error is determined to be ± 5% for all numeric data because of the abrasion test device properties.

Figure 3 shows wear optical microscopy images of 100Cr6 steel extrusion mold specimens after the different treatments at 500 m wear distances. Partly abrasive scars of the presence of adhesive layers can be seen from the traces on the surface of the samples. In Fig. 3 the result of plastic deformation is clearly visible in the wear trace of the untreated sample.

TABLE 3. Wear test results

		Untreated	Hardened	Cryogenic	Tempered
Wear Properties		500 m	500 m	500 m	500 m
21NiCrMo2	Loss of Sample (mg)	0.1521	0.0329	0.0178	0.0255
	Friction Coefficient	0.6321	0.4509	0.2865	0.3209
100Cr6	Loss of Sample (mg)	0.1420	0.0294	0.0167	0.0221
	Friction Coefficient	0.5864	0.3661	0.2367	0.2789

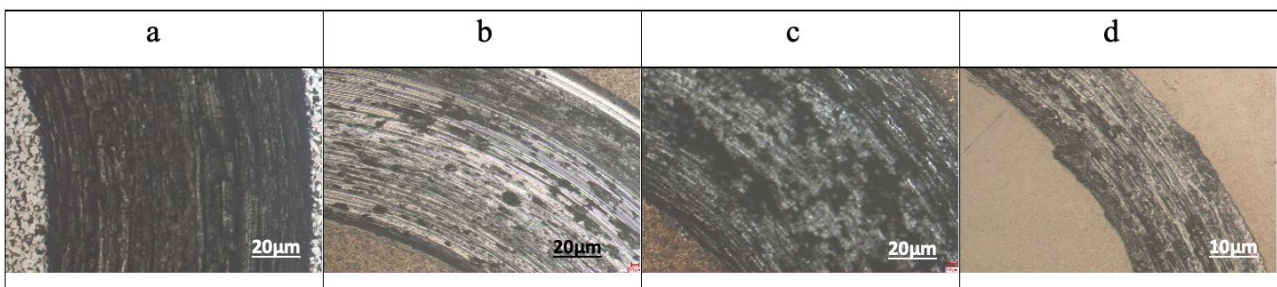


FIGURE 3. Wear trace optical microscopy images of 100Cr6 steel extrusion molds specimens. Distance: 500 m; a) untreated, b) hardened, c) sub-zero, d) tempered samples.

After the different heat and cryogenic treatments, the trace width of the samples decreased. The narrowest wear trace was obtained in the cryogenically processed sample. Cryogenically treated specimens were tempered and the trace of plastic deformation increased due to the decrease in hardness.

The Impact test results of steels 21NiCrMo2 and 100Cr6 extrusion molds after the different heat and cryogenic treatments is given in Fig. 4. The impact energy values were obtained for each treatment condition. The SEM images of the fracture surfaces after the impact tests show that the hardness values were gradually increased in sequence with the application of the different treatments; however the material exhibits a more brittle fracture behaviour.

The untreated steel samples had a highest impact energy. The hardness increased and the toughness (absorbed impact energy) decreased with the application of the different treatments. The notch impact strength values vary in the range from 250 J to 20 J. SEM analyzes of the fracture surfaces were performed and are shown in Fig. 5 and also Fig. 6 for each steel.

Figure 5 shows the SEM images corresponding to the fracture surfaces of steel 21NiCrMo2. These images showed a ductile fracture in the untreated condition. The impact energy absorbed is much more than for any other treatment condition. There was also a circular zone and a brittle-ductile zone separation. Hardening depth can be understood here. It showed brittle fracture after the cryogenic

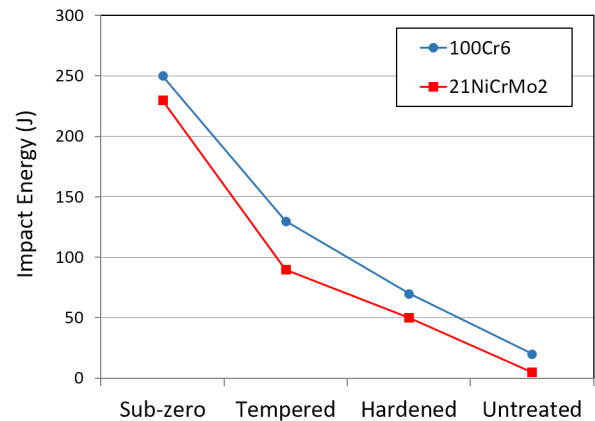


FIGURE 4. Impact test results

treatment. The brittle area has increased. The hardening depth after cryogenic process and tempering showed greater brittle fracture (Fig. 5).

Figure 6 shows that the SEM images of the fracture surfaces after the impact tests of untreated and hardened 100Cr6 steel. This steel in the non-treated condition exhibits a ductile fracture behavior. Therefore, there is slip and more damaged break has been observed. The impact energy absorbed is much more than for the other conditions. A brittle fracture was observed in the sample with the hardened heat treatment. The separation of brittle and ductile shade can be understood from the transition zone in the middle. The cryogenic treatment was less pro-

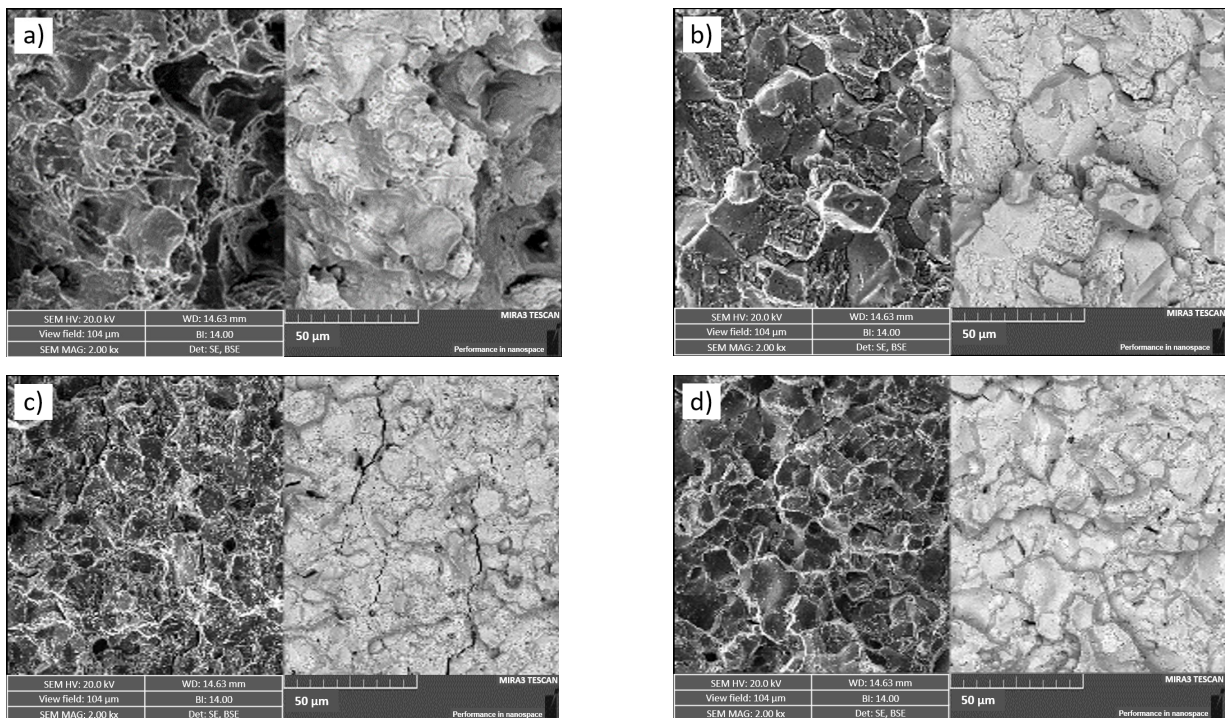


FIGURE 5. SEM images of the fracture surfaces after the impact tests of untreated and hardened 21NiCrMo2 steel extrusion molds specimens, a) untreated, b) hardened, c) sub-zero, d) tempered (scale bar is 50 µm).

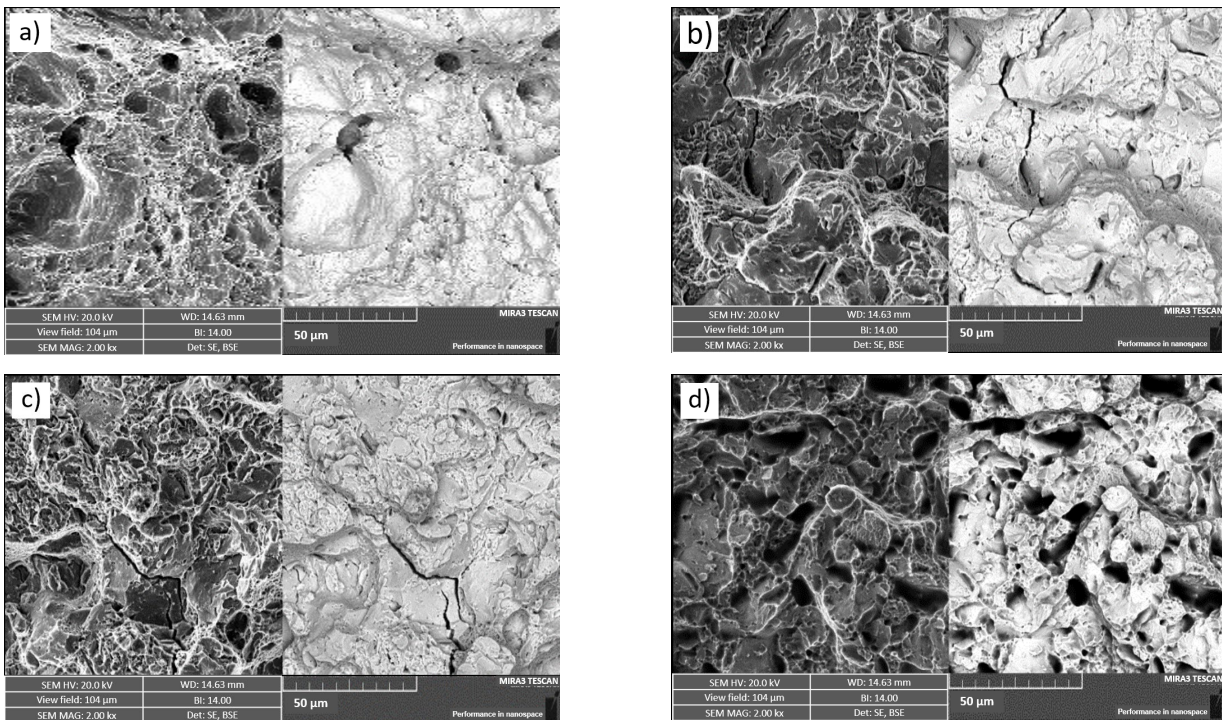


FIGURE 6. SEM images of the fracture surfaces after the impact tests of untreated and hardened 100Cr6 steel extrusion molds specimens, a) untreated, b) hardened, c) sub-zero, d) tempered (scale bar is 50 µm).

nounced in the sample and brittle fracture occurred. The sample under the cryogenic and tempered processes showed no transition region and a brittle fracture was observed (Fig. 5, Fig. 6).

Figure 7 and 8 show the billets produced by casting and a photograph of the extrusion press used in this research. The 100Cr6 steel die without the cryogenic treatment was placed in the extruder and the AA6060 alloy was extruded by applying a rate of 5 mm/s, and extrusion ratios of 30 and 75 (R). The extrusion speed was controlled by measuring the distance at which the punch was displaced in the

horizontal axis-time dependent. Billet temperature was kept constant at 450 °C, shell temperature at 400 °C and the mold initial temperature at 185 °C. AA6060 series alloys have a very high formability, these alloys are poured in the form of billets after the addition of Mg and Si, then they are generally produced by extrusion by pressing through steel molds heated to 400-500 °C (Berndt *et al.*, 2018)

The extrusion molds used in the experiments are shown in Fig. 8. An average of 12 tons of product was extruded with extrusion molds without the cryogenic process. Mechanical deformations, severe abrasions

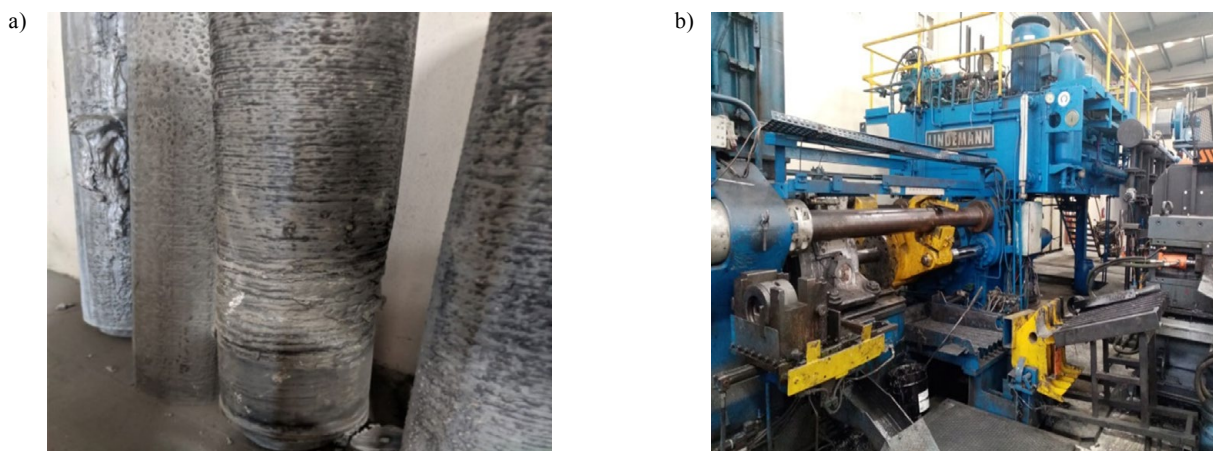


FIGURE 7. Billet of aluminium alloy AA6060 produced by the casting method (a) and a photograph of the extrusion press used in this investigation (b).

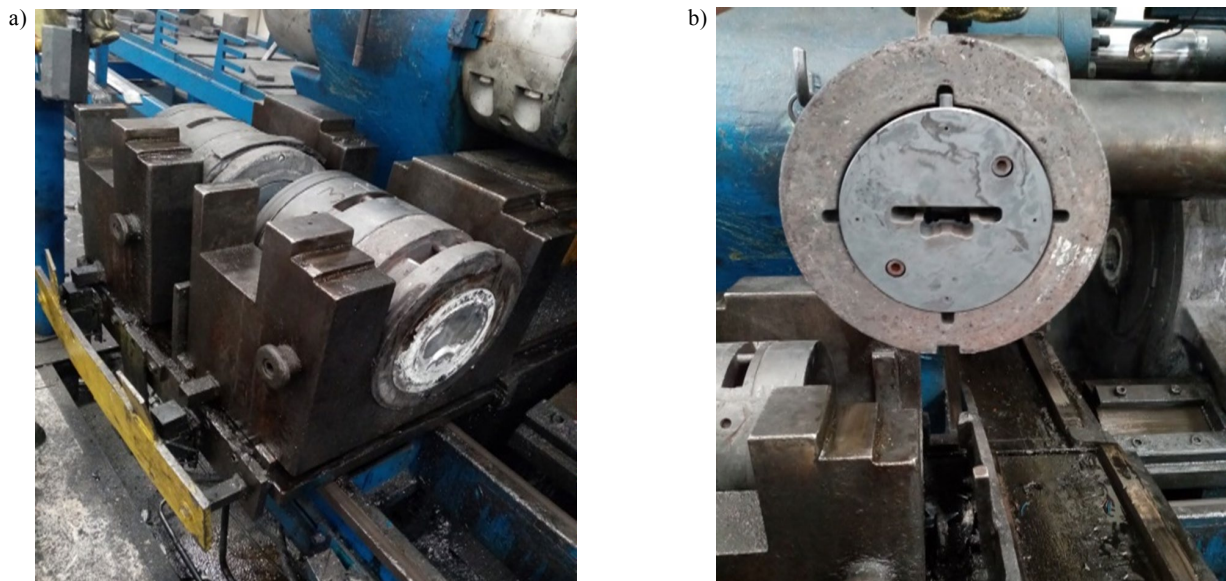


FIGURE 8. The extrusion molds a) isometric view, b) front surface of die and shape of profile



FIGURE 9. Extrusion process of AA6060 Alloy and magnified surface view of the extruded product.

and superficial deteriorations in the extruded products began in steel molds after production amounts of more than 12 tons, and this situation is shown in Fig. 9. An alternative method of increasing die wear resistance without coating or surface treatment is by applying a cryogenic treatment. As it was previously explained, cryogenic treatments are modified cooling treatments undertaken below room temperature by which a phase transformation is promoted (retained austenite to martensite) to increase the hardness and wear resistance of materials subjected to

high abrasion. After the traditional austenitization process applied to the steels, the martensitic phase is obtained by cooling down to room temperature with quenching. However, at this temperature, the high temperature austenite phase to martensite transformation is incomplete, and the steels contain substantial amounts of unstable residual austenite (Büyükfirat, 2019).

From wear optical microscopy images (Fig. 3), it has been concluded that the 100Cr6 steel can be used to produce extrusion molds after being subjected to

the cryogenic process. The wear trough depth in the sample subjected to the subzero treatment show that abrasive scars on the adhesive layers do not occur.

Figure 9 shows that the AA6060 aluminium profile surface. The size and surface properties of the aluminum profile, which is defined as the long product given in Fig. 9, are similar to the extrusion processes performed with hot work tool steel. It can be clearly seen that surface scratches arise from the extrusion die during the extrusion process. This steel die with the cryogenic treatment has a capacity of 18 tons. However, the total extrusion capacity was not reached because of aluminium profile surface disorders observed in this study. This situation arises from surface scratches and roughness defects, not from deformation observed in the product.

Although it is possible that the performance of steels with a cryogenic treatment may decrease over time at production capacities of 18 tons, it has been concluded that it may be effective in the production of low tonnage profiles in the short term.

5. CONCLUSIONS

The deep cryogenic treatment (-120 °C) of quenched and tempered 21NiCrMo2, 100Cr6 steels improves the hardness. The amount of retained austenite is reduced by 75% after the cryogenic process performed after the tempered process for each steel. It is observed that the increase in the amount of residual austenite adversely affects the hardness. The maximum reduction in the amount of residual austenite was observed in the 100Cr6 steel. In the carburized 21NiCrMo2 die steel, residual austenite remained due to the high carbon content in the surface, which has been observed to reduce the wear resistance and fatigue limit of the extrusion dies. As a result, the carbon content of the molds is below 0.7 wt.% C and the quenching temperature (T_q) is lower than the martensite end temperature (M_f), providing a high amount of phase transformation. The steel extrusion dies were subjected to a wear test at a distance of 500 m under a load of 30 N, and the cryogenic hardening process was found to increase the wear resistance. The lowest weight loss (best wear resistance) was 0.0221 mg at a distance of 500 m in the 100Cr6 steel. The notch impact strength values have been brought from the level of 250 J to 20 J. Also SEM analyzes of the fracture surfaces after the impact tests were performed for 21NiCrMo2 and 100Cr6.

As an alternative to existing extrusion dies, it was shown that 100Cr6 steel, provided AA6000 series aluminum extrusion with a capacity of 12 tons. This value is 20-33% lower than the hot work tool steels mold performance. However, it has been concluded that it can be used to extruded some aluminium products which do not request sensitive surface properties.

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