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The effect of a single quenching double tempering process on the mechanical properties of NiCrMo alloyed steel casting

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Abstract. This research aimed to improve the mechanical properties of NiCrMo alloyed steel casting, especially in terms of toughness. Toughness is a combination of tensile strength, yield strength, and elongation. The method used was a multi-heat treatment process involves normalizing, tempering, double tempering, and an additional intermediate process of quenching. The results obtained through the normalizing process followed by single quenching on oil media and double tempering (single quenching double tempering) produced the best-combined result of tensile strength, yield strength, and elongation. The modulus of toughness increased by up to 745 % compared to the as-cast condition from 20 N.mm/mm3 to 149 N.mm/mm3. The best mechanical properties were obtained from tempered martensite microstructure. It is free from rest martensite and secondary carbide.

1. Introduction

There are several known microstructures of steel, such as ferrite, pearlite, bainite, martensite, and austenite. Each structure has different mechanical properties [1,2]. It is possible to get a higher strength from one of these structures. In general, quenching and tempering are effective ways to increase steel strength, which can be achieved mainly through precipitation of fine dispersion of alloy carbides during tempering [3]. The technique is well known to produce the highest level of strength in steel. Martensitic structures are rarely used without undergoing a tempering process because a large amount of internal stress is created due to the transformation process that causes the material to become more brittle [4]. Tempering at low temperatures is enough to reduce this stress significantly without fundamentally changing the basic characteristics of martensite structures. The effect of microstructure on the mechanical properties of low alloy steels has been actively studied by physical metallurgical researchers [5]. The coarse microstructure formed in the ferritic/pearlitic steel hot roll process developed conventionally results in increased ductility, toughness, and strength, which is good. The development of new steel with a better combination of strength, ductility, and toughness has led to the emergence of new types of multi-phase or ultra-fine grained high strength low alloy steel [6].

Applications in the transportation industry demand more economical high-strength steel with good ductility and impact toughness to lighten structural parts [7,8]. Mechanical properties of low alloy steel can be improved by improving austenite grains. Refinement of microstructure on martensitic steels resulting from quenching and tempering can increase steel strength and toughness. The martensitic lath structure is usually stronger, and increasing the toughness of lath martensite low alloy steel is important, especially for industrial applications used at low temperatures. To get a higher combination of strength

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and toughness in low alloy steel, achieved through hardening and tempering, can be done by refining austenite grains by microalloying in addition to cyclic heating, initial microstructure, austenitization temperature and holding time at austenitizing temperatures. These factors are related to each other during the heat treatment process, and the overall effect can be determined by the measurement of austenitic prior to grain size. Besides microalloying and cyclic thermal treatment, the formation of austenite grains from various initial microstructure also becomes an interesting technique adopted in the industry for microstructure refining [9].

Sarikaya et al. found that very high impact energy values could be achieved with double heat treatment, but the strength was slightly lower because of the increase in the amount of austenite held through the double quenching treatment (DQ) followed by low temperature tempering on the base structural steel Fe-Cr-C [10]. Chang et al. studied the effect of double austenitization (DA) treatment on the toughness of Ni-Cr-Mo-V steel with the same strength and found that the increase in toughness of steel treated with DA could be mainly attributed to the lower aspect ratio of carbide [11]. Therefore, steels that are processed with double quenching and subsequent double tempering (DQT) treatments can produce different properties due to different microstructure and morphology as well as the distribution of secondary carbides in steel, the main factors, which improve different mechanical properties.

This research is a continuation of previous research, proposed the use of a single quenching double tempering (SQDT) process [12,13]. The effect of double tempering on the mechanical properties after the normalizing process has been studied on Ni-Cr-Mo alloy cast steel or equivalent to AISI 4340. It was done to improve material toughness through modification of the heat treatment processes. In as-cast conditions, steel casting material has very brittle properties where elongation is only 4%, and impact value is 15 J/cm². Previously the heat treatment process was carried out by normalizing followed by double tempering at a temperature of 650 °C. It was found that steel casting elongation increased up to 20%, and the toughness increased to 142 J/cm² [12,13]. The objective of this research is to study the effects of the additional process of hardening or quenching after normalizing, then followed by a double tempering process on microstructure and mechanical properties of cast steel, primarily with increasing material toughness.

2. Experimental procedure

2.1. Material

The research involved the following steps: For experimental samples, raw materials alloy steel was made of CrNiMo cast, with the composition of the AISI 4340 standard. Steel was cast in the shape of the Y-block sample. Y-block was then cut in a 15x15x12 mm square form. Square samples were used for metallographic examination and Rockwell hardness tests in accordance with ASTM E 18 standards. In addition, the cylindrical sample was also made for tensile tests observing ASTM E 8M standards. Castings were tested for chemical composition using OES (optical emission spectroscopy). The chemical composition obtained is as per Table 1.

		-		1			
С	Si	Mn	Р	S	Cr	Ni	Mo
0,34	0,22	0,70	0,02	0,01	0,87	1,82	0,34

	Table 1.	Chemical	composition	(%)
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The raw material, NiCrMo alloy steel, in as-cast condition has a microstructure with the majority of widmanstaten ferrite, which appears like sharp needles. The other structure is the presence of small amounts of fine pearlite. This steel as-cast NiCrMo has a hardness of 25.4 HRC and a very low elongation of only 4%. Figure 1 shows the microstructure of NiCrMo alloy steel as-cast material.



Figure 1. As-Cast microstructure.

The shortage of elongation makes the steel very brittle. The brittleness comes from the existence of a sharp needle widmanstaten structure.

2.2. Heat treatment

The heat treatment process was carried out by implementing a variety of processes, as shown in Table 2.

No	Sample	Heat Treatment Process		
1	AC	As-cast		
2	Ν	Normalizing 900°C		
3	NST	Normalizing 900°C - Tempering 650°C		
4	NDT	Normalizing 900°C - Tempering 650°C - Tempering 705°C		
5	NSQDT	Normalizing 900°C - Quenching 845° - Tempering 650°C – Tempering 705°C		

Table 2. Heat treatment scheme.

The process for N sample (Normalizing) was done by heating at 900 °C for 2 hours, followed by air cooling. The NST sample (Normalizing Single Tempering) was done by heating at 900 °C for 2 hours, followed by air cooling, then tempered at 650 °C for 2 hours, followed by air-cooled. The NDT sample (Normalizing Double Tempering) was carried out by heating at 900 °C for 2 hours, followed by air cooling, then tempered twice at 650 °C and 705 °C for 2 hours and then air-cooled. The NSQDT sample (Normalizing Single Quench Double Tempering) was carried out by adding a single quench process before the double tempering process as done in the NDT sample. Single quench was done by heating the sample at 845 °C for 2 hours, followed by quench on oil media.

2.3. Mechanical testing and metallographic examination

Mechanical testing, namely tensile testing, was carried out according to E 8M standard, and Rockwell hardness testing followed E18 standard. The metallographic analysis was performed using an optical microscope; the samples etched using 3% Nital. The entire test results were analyzed for effects of the quenching process followed by double tempering treatment compared to other methods according to the various parameters determined prior to the experimental.

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3. Result and discussion

3.1. Microstructures

Figure 2 shows the microstructure observation results. Figure 2 (a) N, shows the microstructure of the sample resulting from the normalizing process. There was no widmanstaten ferrite needle in the microstructure. Instead, the structure was mostly fine pearlite with higher ductility. Figure 2 (b) NST, shows a microstructure resulting from the normalizing process followed by single tempering. White ferrite structures appeared in clusters with higher ductility but lower in strength compared to the N sample. Figure 2 (c) NDT, shows the microstructure of normalized results followed by double tempering. The microstructure had finer grains, whilst the prior austenite grain boundary that appeared on the NST sample was not found, and the ferrite structure was no longer clustered but more dispersed. This makes the sample's ductility higher. Figure 2 (d) NSQDT, shows a normal microstructure followed by a single quench then continued by double tempering. The microstructure appeared as tempered martensite, which was evenly distributed and smooth. There was no longer the presence of ferrite, which made the sample stronger than the NDT sample. However, ductility may have increased because the form of temper martensite had finer grains.



Figure 2. Microstructures (a) N (b) NST (c) NDT (d) NSQDT.

3.2. Mechanical properties



Figure 3. Hardness.

Figure 3 shows the hardness of the samples. The differences in terms of the value of hardness were insignificant. The highest value resulted from the N (Normalizing) process, with a value of 27.2 HRC, and the lowest value came from the NDT (Normalizing-Double-Tempering) process with a value of 22.1 HRC. The hardness value is ascertained based on a combination of the microstructure formed. The N sample had a microstructure of mostly fine pearlite, which was harder, whilst the presence of ferrite in the NDT sample made it softer.



Figure 4. Tensile test.

Figure 5. Elongation.

Figures 4 and 5 show the results of tensile testing data. Tensile strength in as-cast conditions was 786 MPa, with 4% elongation increasing after normalizing to 1051 MPa with elongation up to 8.8%. The result shows that the normalizing process can significantly improve the mechanical properties of cast steel by transforming the widmanstaten ferrite structure to fine pearlite [14]. This increases both elongation and strength simultaneously.

The various multiple heat treatment processes after normalizing, namely single tempering, double tempering, and quench double tempering, increase ductility for each sample. The double tempering process produced a finer grain structure [15-18]. In the double tempering process, the first tempering process reduced the hardness by transforming martensite to tempered martensite but can produce brittle secondary carbide from the steel alloy. First, tempering produced martensite from the rest austenite, resulting from the quenching process [14]. The second tempering process tempered the martensite, reducing brittleness and at the same time, producing the fine grain. The NSQDT process increased elongation up to 19.8%.



Figure 6. Tensile test curve.

Figure 6 shows the tensile test curve. The original curve from the test machine was incorrect because there was an error when recording the elongation associated with the slip in the sample gripping. Figure 6 shows the correction done. From the curve, the modulus of toughness can be calculated. This value measures the ability of the material to absorb energy, which is represented by the area under the curve. The formula to calculate the modulus of toughness is as per (1) below [19]:

$$U_T = \frac{so+su}{2}e_f \tag{1}$$

UT Modulus of toughness (J/m³) or (N.mm/mm³)

- S_o Yield strength (MPa)
- S_u Ultimate tensile strength (MPa)
- e_f Elongation at fracture



Figure 7. Modulus of toughness and tensile test curve.

Figure 7 shows the results of the calculation for the modulus of toughness for all the samples. The highest value was produced by the NSQDT sample. The NSQDT process produced CrNiMo alloy steel materials with the highest modulus of the toughness of 149 N.mm/mm³. The toughness is a combination of the tensile strength, yield strength, and elongation. The high value was the result of the quenching process, followed by double tempering. The process formed a microstructure of tempered martensite with fine grains, free from residual "brittle" martensite and free "brittle" secondary carbide at the prior austenite grain boundary.

4. Conclusion

The results of this study showed the NSQDT (Normalizing Single Quenching Double Tempering) process had the best combination of mechanical properties with good strength, yield strength, and elongation. It contributed to the toughness of NiCrMo steel. The modulus of toughness increased by up to 745 % compared to the as-cast condition from 20 N.mm/mm³ to 149 N.mm/mm³.

The NSQDT process formed a microstructure of tempered martensite with fine grains, with high tensile strength and good mechanical properties. The second tempering process eliminated the martensite, after the first tempering process that made it tougher.

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