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Exploring the Effect of Long Immersing Time and Copperized Cooling Aisi 1006 on Micro Structure and Hardness Properties

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Abstract: Copperizing is the addition of copper using a hot-dip technique. The addition of copper is done because it can increase the hardness of low carbon steel, by reducing the grain size. This study aims to determine the effect of immersion and cooling time on the microstructure and hardness properties of copperized AISI 1006 and its strengthening mechanism. Variations in the immersion time used were 10 minutes, 20 minutes and 30 minutes as well as normal and fast cooling (quench). Tests carried out in this research were spectrometric tests to analyze elements, x-ray diffraction tests to observe the phase transformations formed, metallographic tests to observe changes in microstructure, EDX SEM tests to observe topology and distribution of copper atoms, and microhardness tests to analyze hardness. phase obtained from the test sample. The results showed that there had been changes in the microstructure and hardness properties. The microstructure obtained on normal cooling and immersion time of 10 minutes; 20 minutes; and 30 minutes is ferrite, ferrite, and pro-eutectoid ferrite. While the microstructure obtained on quench cooling and immersion time of 10 minutes; 20 minutes; and 30 minutes is ferrite, martensite and bainite. The maximum hardness value obtained by quench cooling and immersion time of 20 minutes is 309.97 HV due to the formation of a martensite microstructure. The strengthening mechanism that occurs in AISI 1006 with the copperizing method is the reduction of ferrite grains for immersion times of 10 minutes normal cooling and quenching, and 20 minutes normal cooling. While the other test samples experienced changes in microstructure.

Keywords: AISI 1006, Copperizing, hardness, microstructure, copper.

INTRODUCTION

Steel is an Fe-C alloy that always develops from each period, starting from carbon steel (low, medium and high), non-steel (carbon content above 2%), and alloy steel (Jaypuria, 2009). The basis of the development of alloy steel due to the basic nature of steel that is easily corroded, low ductility and to obtain some other steel properties such as formability, machinability. Many alloys have been developed to obtain better steel, especially in micro-alloying by producing high-strength low-alloy steel to balance strength and ductility (Skobir, 2011).

Copper has excellent thermal and electrical conductivity, good strength and formability, excellent corrosion and fatigue resistance, and is generally non-magnetic (CDA, 2010). The addition of copper to low carbon steel can increase strength and also improve hardness properties, as well as reduce the area of bainite and martensite structure formation in the aging treatment (Wilson, 1990).

Not only with alloying elements, but by giving treatment to the steel it can bring the desired mechanical properties (Thelning, 2000). Various kinds of treatment can be given to the steel such as quenching, normalizing, perfect annealing, and so on. When steel has been given treatment and alloying elements, the strengthening mechanism due to that process can show the properties that arise (Callister, 2007). Therefore, a study was carried out on the effect of copper on the microstructure and hardness properties of plain carbon steel combined with the copperizing method and variations in cooling. The research carried out also analyzed the strengthening mechanism of steel through the copperizing process.

RESEARCH METHODS

Research Methods

Before the research was carried out, sample preparation was carried out by cutting and rubbing the surface of the material to be dyed to obtain the desired results. After the material preparation process is complete. The plate material is put into the muffle furnace, while the copper is melted in the vacuum furnace. The method of making test specimens is done by immersing the plate in molten copper. Samples that have been dyed are then varied by cooling. The sample results will be tested using several test methods to analyze the effect of immersion time and cooling on the sample. The analysis carried out was chemical composition analysis using OES testing, XRD analysis, microstructural analysis using an optical microscope, and SEM EDX analysis using the FEI INSPECT S50 instrument, and hardness analysis was carried out with the Wilson Instrument micro hardness testing machine and then compared with the hardness range according to the control sample. .

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Materials

The starting materials used in this study were AISI 1006 low carbon steel, 99.99% copper (Cu) and ASTM E407 etching material. Low carbon steel is obtained from scrap metal in the form of plates with a thickness of 3 mm. Steel was chosen as the base metal in this alloy because it contains at least 70% Fe. In addition, the low carbon content in steel is chosen to eliminate the carbon decarburization process. This low carbon steel was obtained from the metallurgical laboratory of the Department of Materials and Metallurgical Engineering ITS. Analyzing the microstructure of the resulting alloy, it is necessary to use an etching fluid. In this study, the ASTM E407 etching material was used, which is a mixture of HNO3 and methanol. Etching fluid obtained from CV. Persada Scientific Resources.

Tools (Instrument)

The tools used in this study were copper furnaces, muffle furnaces, graphite crucibles, saws, 250 ml measuring cups, beaker glasses, scales, sandpaper, chainsaws, hardness test kits, Optical Emission Spectroscopy (OES), XRD, grinding machines & polishing, and metallurgical microscopy. This copper furnace is used to melt copper which will be used as an alloy. This furnace is homemade, using Fe-Cr wire as a conductor of heat from electricity. The vacuum furnace used is shown in Figure 3.2. The crucible used for smelting copper and for the dyeing process is a graphite crucible. The crucible size used is 150 grams. Cutting materials and alloy results, saws and chainsaws are needed. A measuring cup with a capacity of 250 ml is used to measure ingredients when preparing etching solutions. The container for mixing the etching material is used in a beaker glass. Measuring the mass of copper to be melted uses an electric scale with an accuracy of up to 0.01 gram. Sandpaper with grades 80 to 2000 is used for grinding specimens in metallographic tests.

This research used toho branded sandpaper. To facilitate grinding and polishing in metallographic tests, this study also used grinding and polishing machines manufactured by the material and metallurgical engineering department. The microstructure contained in the alloy material can be examined using a metallurgical microscope with the Olympus brand with a magnification of up to 500x. The topography of the test sample can be analyzed using SEM EDX analysis with the FEI INSPECT S50 instrument. Analyzing the hardness of the resulting alloy, then using the Micro Hardness Tester Wilson Instrument machine contained in the material and metallurgical engineering department. Meanwhile, to find out the composition of the alloy, the OES tool was used which was found at the Surabaya State Polytechnic of Shipping. X-Ray Diffraction test equipment is used in this specimen to determine the crystal system, crystal structure, and phases contained in the alloy. The XRD machine used is PAN Analytical branded in the material and metallurgical engineering department.

Figure 1. Vatembagaum Furnace (from the front side)

Experiment Procedure

Specimen preparation was carried out by cutting a plate of 3x5x0.3 cm, then rubbing the surface of the plate using grade 80 rubbing paper. Weighing the copper with an electric scale of 1.8 kg according to the capacity of the crucible used. Smelt copper in a vacuum furnace to a temperature of 1080 oC by injecting Ar gas to minimize decarburization. This is done together by heating the plates in the muffle furnace to a temperature of 900 oC. Then dip the plate in molten copper with variations in calup time for 10, 20 and 30 minutes. After that, provide cooling variations on the sample with normal and fast cooling (quech) in water media. These results are followed by several tests to obtain data that can be analyzed.

Testing Process

This research conducted four tests. Composition testing by OES, Metallographic testing to see the microstructure. Hardness test to determine the hardness value. XRD test to determine the phase formed.

1. OES Testing

OES testing, Optical Emission Spectroscopy, is a test that involves applying electrical energy in the form of a spark generated between an electrode

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and a metal sample, whereby the vaporized atoms are brought into a high energy state called plasma discharge. The excited atoms and discharge plasma create a unique and specific emission spectrum for each element, as shown in Figure 3.4. Thus one element produces many characteristic emission spectral lines.

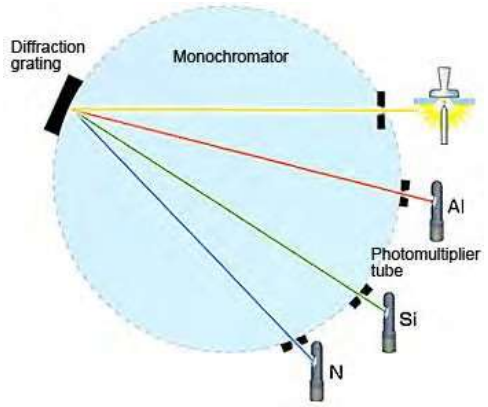

Figure 2. OES working principle (shimadzu.com)

Therefore, the light produced by the emission of electrons can be said to be a collection of spectral lines produced by the elements in the sample. This light is split by a diffraction grating to obtain the emission spectrum of the sample target. The intensity of each emission spectrum depends on the concentration of elements in the sample. The detector calculates how much spectrum is obtained from each element and the intensity of each spectrum to carry out qualitative and quantitative analysis of elements.

This test was conducted to determine the composition of the research sample. OES testing in this study was carried out at the Surabaya Shipping State Polytechnic, by shooting 3 times.

2. XRD Testing

XRD (X-ray Diffractometer) is a material testing tool that is usually used to identify elements or compounds (qualitative analysis) and determine composition (quantitative analysis). The analysis carried out relates to other measurement tools such as SEM or TEM. Observations with a microscope will explain how the phase distribution is identified based on the XRD results.

XRD testing utilizes the diffraction of X-rays. In general, the working principle of XRD can be seen in Figure 3. The high voltage generator functions as an X-ray source power generator in the x-ray tube section. The compressed powder sample is placed on a container that can be positioned. Then the X-ray beam is shot at the sample and is diffracted by the sample, entering the chopper. The X-ray diffraction intensity is captured by the detector and translated into a curve.

X-ray diffraction analysis was carried out with the aim of identifying the phase transformation that is formed in the Fe-Copper alloy that has been formed during the copperising process. The possible phase which will be copper is Ferrite (α) this is due to the influence of the very low amount of carbon. In this study, highscore software and JCPDF win were used to determine and analyze the peak results of XRD

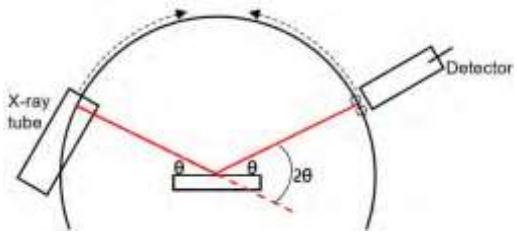

Figure 3. XRD Work Scheme (Moore and Reynolds, 1997)

The working principle of XRD testing is that when a material is exposed to X-rays, the intensity of the transmitted light is determined lower than the intensity of the incident light. This is due to absorption by the material and also scattering by the atoms in the material. Some of the scattered X-ray beams cancel each other out because the phases are different and some are mutually reinforcing because of the same phase. These mutually reinforcing X-rays are known as diffracted beams. XRD testing was carried out at the Materials Characterization Laboratory, Department of Materials and Metallurgical Engineering, ITS using a PAN Analytical XRD machine. The filament used is copper (Copper) with a current of 30 mA, and a voltage of 40 kV. The tested sample has a maximum dimension of 3 mm in thickness.

3. Metallographic Testing

Metallography is a testing method to view metal structures on a micro scale. This is done using an optical microscope and an electron microscope. The structure or image of the metal that is visible through observation with a microscope is called microstructure. This figure shows the scope of the microstructure of metal which is generally observed with a microscope. In this study the magnification used was 5-50 times the magnification of the objective lens.

The microstructural investigation ranged from 10-6 cm (the limit of the ability of the electron

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microscope to 10-2 cm the upper limit of the ability of the human eye). Although the scope of this metallographic observation covers a large area (10-6 - 10-2 cm), the objects of observation that are usually used are 10-5 cm or an order of magnification of 5,000 - 30,000 times for electron microscopy and 10-3 cm or an order of magnification 100– 1000 times for an optical microscope.

Metallographic observations are based on the difference in the intensity of the reflected light on the metal surface that enters the microscope so that different images occur (dark, slightly bright, bright). If the metal surface that has been refined (polished) is then etched with a chemical solution, then metal surface will be dissolved. Different microstructures will dissolve at different speeds, leaving surface marks with different angular orientations. Thus, if a beam of light is applied to the metal surface that has been tested, the light will be reflected according to the angular orientation of the surface of the area affected by the reflection.

The purpose of metallographic testing in this study was to look at the microstructure formed in the alloyed specimens. The visible microstructure will be compared with the alloy phase diagram. This observation was carried out to see the effect of the alloying elements on the phase diagram and its microstructure. This observation was carried out using an Olympus BX51M-RF optical microscope, while the etching solution used was in accordance with ASTM E-407, namely nital. The incident beam mechanism in metallographic testing.

4. Scanning Electron Microscope Tests

This test was carried out to observe the topography of the cross-sectional surface of the copperizing test specimen, and the distribution of copper diffused into the AISI 1006 steel. The principle is the same as metallographic testing, but for this SEM test you can see the distribution of copper. The type of machine used for SEM EDX testing is the FEI INSPECT S50 in the ITS material and metallurgical engineering department. In this test, we will look at the topography so that a secondary electron detector is used with parameters that are edge observations with a magnification of 1000x.

Microhardness Testing

Microhardness testing is a form of hardness test. Microhardness is needed to ensure the hardness of a phase, so that the hardness value of each phase that appears in the microstructure can be ascertained. The microhardness test is carried out using the Vicker hardness, so it has a small scope for relatively hard materials. The microhardness test was carried out using a Wilson Instrument machine and was carried out in accordance with the ASTM E92 standard. The loading given is 1 kgf. From the tests carried out, the diameter of the indentation was obtained which was then entered into equation 3.2.

$$HV = 2P \sin\frac{\alpha}{2} = 1{,}8544 \frac{P}{d^2} \qquad (3.2)$$

Where P is the loading in kgf units, D is the average diameter of the indenter in μm units.

RESULTS AND DISCUSSION

This study aims to determine the effect of immersion time and cooling variations on changes in the microstructure and hardness properties of AISI 1006 steel. The cooling variations provided are in the form of normal and fast cooling (quench) in water media. The results of the study explain several data such as thickness, composition, XRD spectra, and the microstructure and hardness of the samples. The results of the study sample were compared with the control sample adjusted for the research method

The results of macro observations on the samples showed a reduction in thickness. Thickness reduction that occurs is proportional to the immersion time. The previous table shows that the longer the immersion time and also the cooling given, the effect on the thickness of the sample in terms of incorporation (Thelning, 2000). The thickness reduction found can be related to the amount of copper deposited on the exposed surface. Each test sample has copper deposits on its surface which are shown in orange. If the length of immersion time will reduce the thickness of the sample, while the length of immersion time cannot increase the copper deposit. Thickness reduction that occurs due to erosion. Erosion that occurs due to molten copper. This erosion can occur due to several factors ranging from temperature, time and amount of molten copper. Due to the effect of the long exposure time on the melting temperature of

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copper with a greater amount of copper than the test sample. Called erosion due to a significant reduction in thickness. The reduced sample dissolves in the molten copper (AWS, 2007). So if the copper layer/deposit gets thicker it can cause lower tensile strength (Sharma *et al*., 2014). If area reduction occurs, it can increase the hardness (Thelning, 2000).

The results of the XRD test show peak brodening of the phases that have the highest intensity. This peak broadening shows graphically the addition of elements which results in reduced grain from figures. There was a 2θ shift in the XRD results, this could be due to the atoms diffusing into the test sample. Diffusion that occurs does not change or form a phase. In addition to the 2θ shift, there was also a strain (micro strain) of the FWHM obtained after the XRD test. The strain that occurs is getting bigger in proportion to the addition of the elements being made. Copperizing means microalloying, with copper having the effect of shrinking grains (Tohidi, 2012). According to the ASM Handbook (1990) copper dissolved in steel can reduce grain size, even adding it at a certain level can reduce grain size to an extreme, that's why copper is often referred to as an ultra-grain refinement agent in steel. It was also explained that the reduction in ferrite grain size due to the addition of copper was due to the presence of copper which inhibited austenite recrystallization and inhibited the transformation of austenite to ferrite. The addition of copper also affects hardness (Wilson, 1990).

The results of micro observations, obtained a different microstructure. The more elements added, the smaller the grains will be and they will not be different from the microstructure of the sample without treatment (Tohidi, 2012). In Figure 4.4 (d) the proeutectoid ferrite microstructure is obtained. This proeutectoid is a mirostructure in which ferrite-α nucleates with an austenite-γ matrix. Can occur if the steel is heated at a temperature of 1200 oC for 30 minutes, then cooled down and is in the α+γ zone. Proeutectoid or α-primary can occur in steel with an element of 0.12% C (Tamura, 1988). In Figure 4.5 (c) has a martensitic structure, and Figure (d) has a bainite structure. This can happen because the addition of alloy can lower the temperature of Ms and Bs. The structure of martensite and bainite can occur if previously heated at a temperature of γ. Then it is cooled until it reaches Ms and Bs/. This is what makes the austenite deformed. This structure can be formed even though it has 0.06% C (Wilson, 1990).

The mapping results show that the distribution of copper is only in the surface layer. However, in samples under normal conditions some of the copper goes into Fe. The entry of copper into Fe is not too deep and in small amounts, and it is penetrated at the grain boundaries. It was done by Xu *et al*., that some copper can diffuse along the ferrite grain boundaries and in the ferrite grains. If it is related to the OES results which show that copper levels seem to fluctuate, so it does not indicate the significance of diffused copper.

The hardness value obtained is due to the treatment given. Normalising treatment makes the structure more homogeneous and responds well to violence (Callister, 2007). While the quench treatment makes the structure inhomogeneous. The quench treatment tends to create a martensitic structure and is very small for bainite to occur. The hardness value for the bainite structure tends to be half that of the martensitic structure and above the hardness value of the pearlite structure (Thelning, 2000). When compared to the hardness that occurs due to grain reduction, the hardness value of martensite is far greater (Juristry, 2013). Therefore, the highest hardness value was obtained during the quench cooling treatment and 20 minutes of immersion time with a martensite structure.

CONCLUSION

Based on the results of data analysis and testing conducted in this study, it can be concluded that:

1. The copperizing method with normal cooling and immersion time parameters of 10 minutes has a ferrite microstructure with a content of 0.06%Cu, 20 minutes of a ferrite microstructure with a content of 0.03%Cu, and 30 minutes of a microstructure of proeutectoid ferrite with a content of 0.06% Cu.
2. The copperizing method with rapid cooling (quench) and immersion time parameters of 10 minutes has a microstructure of ferrite with a content of 0.05%Cu, a microstructure of martensite with a content of 0.44%Cu in 20 minutes, and a microstructure of bainite with a content of 0.44%Cu for 30 minutes. 04%Cu. The deviation of %Cu at 20 minutes of immersion time was due to the level of cleanliness before the OES test.
3. The maximum hardness value obtained by fast cooling and immersion time of 20 minutes is

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309.97 HV because the microstructure formed is martensite.

4. The strengthening mechanism that occurs in AISI 1006 with the copperizing method is the reduction of ferrite grains for immersion times of 10 minutes normal cooling and quenching, and 20 minutes normal cooling. While the other test samples experienced changes in microstructure.

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