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# EFFECT OF ANNEALING ON MICROSTRUCTURE AND HARDNESS OF FeNiCo ALLOYS SYNTHESIZED BY MECHANICAL ALLOYING

**Sutrisna [1) ✉], Didit Setyo Pamuji[1)], Angger Bagus Prasetiyo[1)], Ismail Zulpria Ababil[1)], Ihwanul Aziz [2)]**

[1)] **Department of Mechanical Engineering,** Institut Teknologi Nasional Yogyakarta,
Jl. Babarsari Caturtunggal, Depok, Sleman,
Yogyakarta 55281
sutrisna@itny.ac.id

[2)] **Research Center for Accelerator Technology**, National Reserch and Innovation Agency,
Jl. Babarsari Caturtunggal, Depok, Sleman,
Yogyakarta 55281

***Abstract***

*Alloys based on Fe-Ni-Co are categorized as special nickel-based superalloys with braod application as magnetic sensors in electric motors, recording devices as well as for vehicle engine parts. The purpose of this study was to determine the physical and mechanical properties of Fe-Ni-Co materials synthesized by mechanical alloying method, then subjected to annealing temperature variation i.e 800°C, 900°C, and 1000°C for 1 hour. Test methods carried out by XRD-spectra to identify the phase, SEM and EDS mapping to determine the microstructure and composition together with micro hardness test to represent the mechanical properties of this FeNiCo alloys. The results of the XRD test showed that Co atoms and some Fe dissolved into the Ni crystal lattice tended to form a solid solution of γ-Ni(Fe,Co). Microstructural observations at 900 °C after milling for 16 hours showed a fine and homogeneous grain structure. The highest hardness value was 421.5 kg/mm$^2$ at 16 hours milling. The Fe-Ni-Co alloy exhibited a homogeneous microstructural distribution with a fine grain structure and high densification.*

***Keywords:*** *FeNiCo Alloys, Annealing, Mechanical Alloying, Milling, Microstructure, Hardness.*

## 1. INTRODUCTION

Alloys based on iron, nickel and cobalt are of particular interest for the development of special steels, Ni-based superalloys and special materials in composites. This combination of alloys is promoted for magnetic sensors, recording devices, and integral schemes[1], [2] Furthermore, the Fe-Ni-Co alloy is the basis for forming high entropy alloy components. This is because this alloy is a solid solution alloy at high temperatures of the Fe-Ni-Co alloy[3], [4]. Therefore, research on the phase balance in this alloy is very important to optimize the crystallization process in controlling the structure and material properties at high temperatures.

Based on the literature review shows data in the high temperature region is limited to work but most studies focus on the solid-state phase relationship at low temperatures [5]. In addition, research conducted by C. Wang et al [6].describes the phase boundaries of γ (a solid solution based on Fe, Ni, Co with FCC structure) and αFe (based on Fe with BCC structure).

*Corresponding Author*:
✉ **Sutrisna**
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Furthermore, this alloy steel has also been reported to have many other attractive properties such as high strength and toughness at room and low temperatures [7], good fatigue resistance [8] and good resistance to oxidation at high temperatures as well as the ability to age harden and absorb energy upon impact [9].

The application of Fe-Ni-Co steel in both the industrial and automotive fields is very broad, besides that it is also used as a base material for high-entropy alloys and high-temperature alloys. This is supported due to its outstanding properties such as mechanical properties, formability, impact toughness, and corrosion resistance [10]. Recently, that Fe-Ni-Co powder alloy by mechanical alloying method is used as a base material for vehicle parts [11] and also as a base in high and medium entropy alloys [12] and composite reinforcement in heavy tungsten alloys [13] .

More specifically related to this Fe-Ni-Co mechanical alloy with the same (equatomic) and different (non-equatomic) elemental content still requires further investigation, especially on changes in the microstructure and mechanical properties of the material at high temperatures. This research focused on non-equatomic Fe-Ni-Co powder alloy materials after milling for 4 hours, 8 hours and 16 hours for their physical and mechanical properties after annealing at temperatures of 800, 900 and 1000 $^{O}$C. The outcomes of this research may have significant implications for the development of new materials with unique properties. These materials could find applications in diverse industries, potentially opening up new opportunities in engineering and various industrial sectors.

## 2. METHODOLOGY

The materials used in this study were powders of the elements Fe, Ni, and Co, with particle sizes between 1 - 50 microns. Its chemical composition is 25% Fe, 50% Ni and 25% Co. The method used in this research is to mix the elements first with the percentages mentioned above. Furthermore, the elements were milled using the mechanical alloying method for 4 hours, 8 hours and 16 hours at 300 rpm. 10:1 ratio between ball and powder. Moreover, the milled powder is compacted with a pressure of 5 tons within 5 seconds to be a green compact. Then the green compact was annealing at 800$^{o}$C, 900$^{o}$C, 1000$^{o}$C and held for 1 hour. Tests carried out with full term of XRD spectrum were used to characterize the powder phase and full term of SEM (SEM-EDS) mapping was used to determine the microstructure and composition. Furthermore, the hardness test was carried out with micro-Vickers using a load of 200g for 5 seconds.

## 3. RESULT AND DISCUSSION

### 3.1. Characterization of Powders

In this study, XRD testing was carried out to identify a material in the form of Fe-Ni-Co powder after grinding for 4 hours, 8 hours and 16 hours. In addition, it is also used to analyze the material phase that occurs during the milling process. Data from the XRD test results show that the peak height changes significantly but the lattice width does not change, it indicates specific structural changes in the material. As shown in Figure 1. that at the milling time of 4 hours and 8 hours the peak elements that occur are very high. The highest peak on the Ni element overlaps with Fe, then the other peaks are dominated by the Co-oxide element which is seen scattered in many places because of chemical similarity. The distribution of elements in more detail is shown in Figure 2 and Figure 3. At the end of 16 hours milling, the overlapping elements of Ni and Fe form a Fe-Ni alloy (as shown in Figure 4). Fe-Ni alloy is a very strong alloy and is capable of making high quality steel [14]. In principle milling 4 hours to 16 hours has the same peak shape, but the peak height decreases. This peak decrease is due to the mechanical alloying process so that the elemental grains become small and fine. In addition, Co atoms and some Fe dissolved into the Ni crystal lattice will tend to form a solid solution of γ-Ni(Fe,Co) [15].

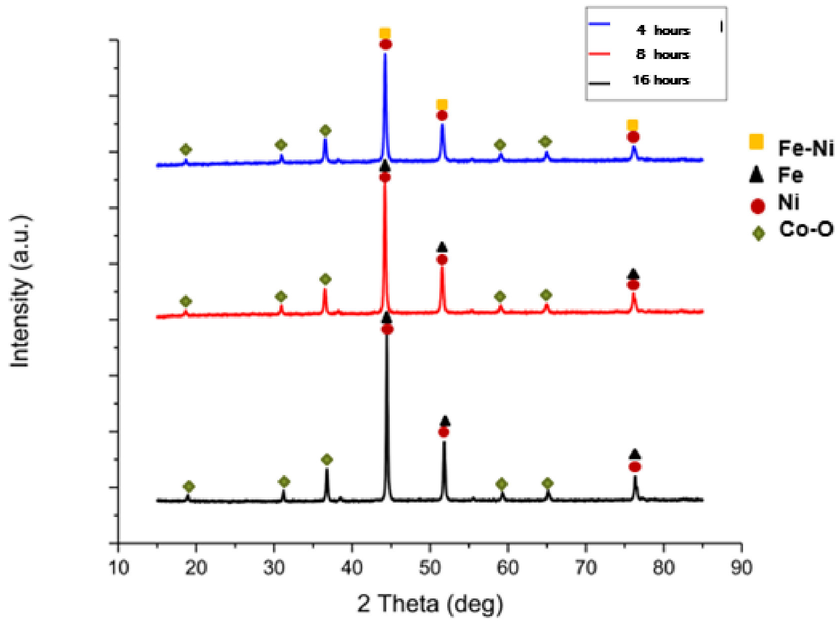


**Figure 1.** XRD-spectra with milling variations of 4 hours, 8 hours and 16 hours

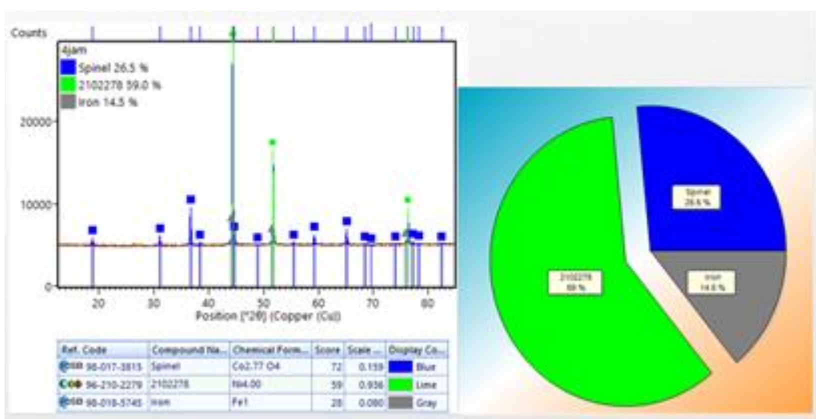


**Figure 2.** Detailed analysis of XRD-spectra at 4 hours milling

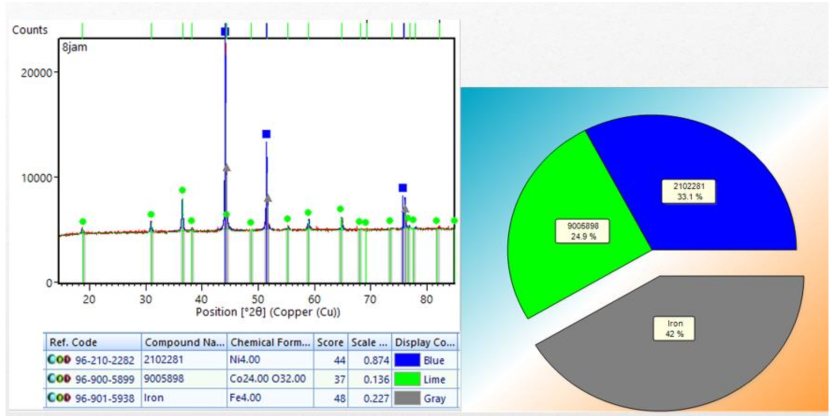


**Figure 3.** Detailed analysis of XRD-spectra at 8 hours milling

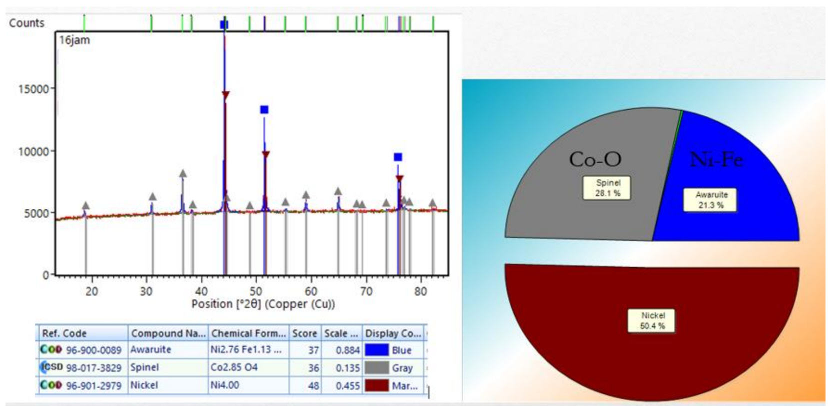


**Figure 4.** Detailed analysis of XRD-spectra at 16 hours milling

### 3.2. Microstructure Characterization

The results of the microstructure test of the Fe-Ni-Co alloy was carried out by observing the test object using the Scanning Electron Microscopy (SEM) method. The alloy was milled with a variation milling time of 4 hours, 8 hours, 16 hours, then heated at 800$^{O}$C, 900$^{O}$C and 1000$^{O}$C with 1 hour holding time. In Figure 5. shows the microstructure that occurs in the Fe-Ni-Co alloy after milling for 4 hours, 8 hours and 16 hours and annealing to a temperature of 900$^{O}$C. The results of microstructure observations show that there are three areas that have the dominant color. The white color indicates the element Ni, gray represents the element Fe and the element Co is shown in blackish gray.

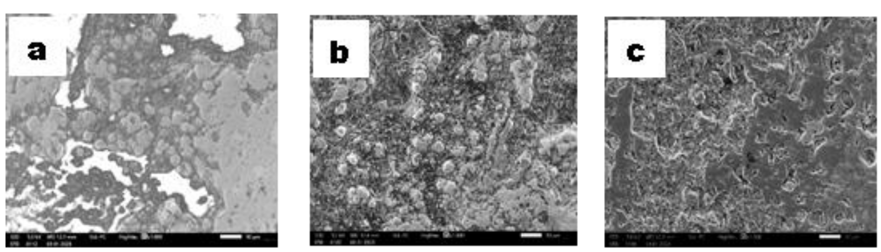


**Figure 5.** Microstructure with variations in milling a. 4 hours, b. 8 hours and c. 16 hours after 900$^{O}$C annealing

Apart from being indicated by these colors, it is also clear that the elements which are very dominant in the Fe-Ni-Co alloy, namely Fe and Oxides will form iron oxide ($Fe_2O_3$). The results of observations of the microstructure of the Fe-Ni-Co alloy with a milling time of 4 hours showed that the distribution of elements was uneven and the grain size of each element was also imperfect shown in Figure 5.a. This is because the milling time of 4 hours is not enough for the elements of Fe, Ni and Co to become small and homogeneous so that the composition of the elements is still separated and agglomerates occur in certain areas. After the milling time was increased to 8 hours, the composition of the elements became more perfect and the grain size began to decrease. At the end of 16 hours of milling, it is clear that the composition of the elements is perfectly dispersed and the grain size becomes finer. This shows that in milling 16 hours at 900°C annealing occurs in the form of a solid solution [16]–[18].

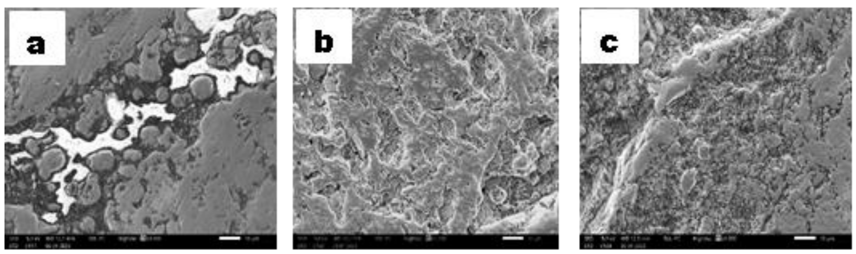


**Figure 6.** Microstructure with annealing variation at temperature a. 800 $^{O}$C, b. 900 $^{O}$C dan c. 1000 $^{O}$C

In figure 6. showed changes in microstructure after heating with temperature variations of 800 $^{O}$C, 900 $^{O}$C dan 1000 $^{O}$C. There was a very significant structural change from that temperature. At 800 $^{O}$C a solid solution has not yet occurred, it can be seen that the elements are still clumping in certain areas. In addition, the distribution is not evenly distributed. This shows that the temperature of 800 $^{O}$C is not sufficient for changes in elemental forms to occur

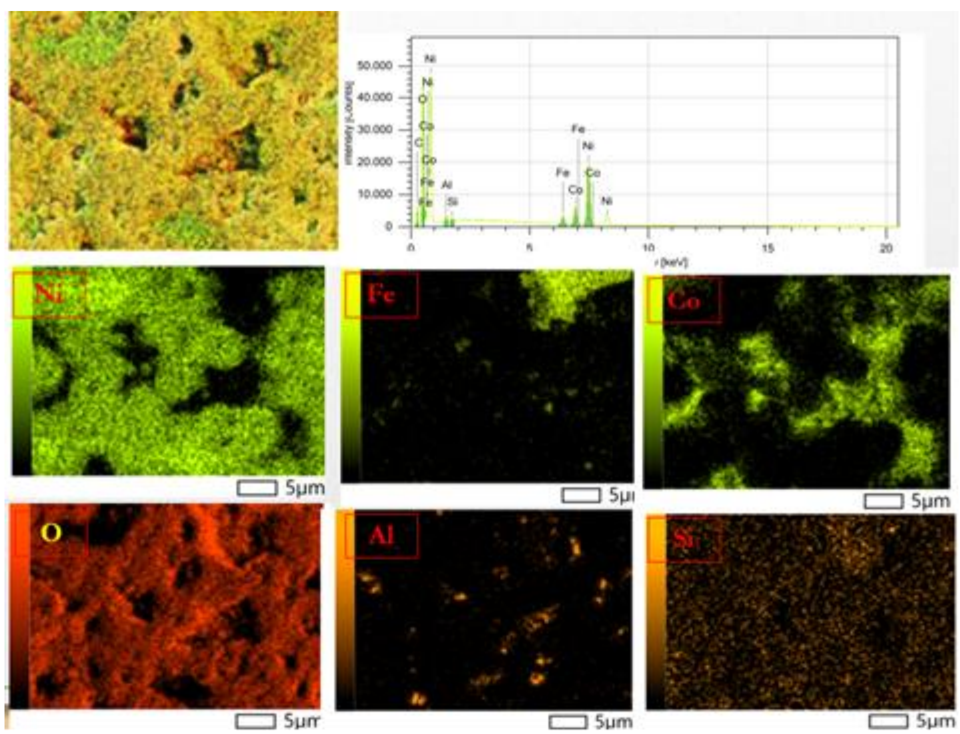


**Figure 7.** EDS mapping on Fe-Ni-Co alloys after 800 $^{O}$C annealing.

After the temperature was increased to 900 $^{O}$C there was a significant change in shape and structure, the elements were spread evenly and a solid solution occurred. However, after the temperature was raised again to 1000 $^{O}$C, a very large oxide with Fe appeared to form iron oxide ($Fe_2O_3$).

### 3.3. EDS Mapping Analysis

In Figure 7. shows the distribution of Fe-Ni-Co alloying elements after annealing temperature of 800 $^{O}$C, agglomeration occurs in certain areas. The element Ni has a very high distribution, as well as the element oxygen which is also very visible in the alloy. This happens because the Ni element has a high composition, which is 50% of the alloying elements, In addition, the process of mixing alloys and printing specimens is carried out in free air that contamination with outside air is very large. The phenomenon can be said that the Fe-Ni-Co alloy after being heated to 800 $^{O}$C has not yet formed a solid solution and after the temperature is raised to 900 $^{O}$C the distribution of the elements is even and mechanical alloying occurs, as shown in Figure 8. In addition, there is a very high contamination between iron (Fe) and oxygen (O) that it forms an iron oxide phase ($Fe_2O_3$).

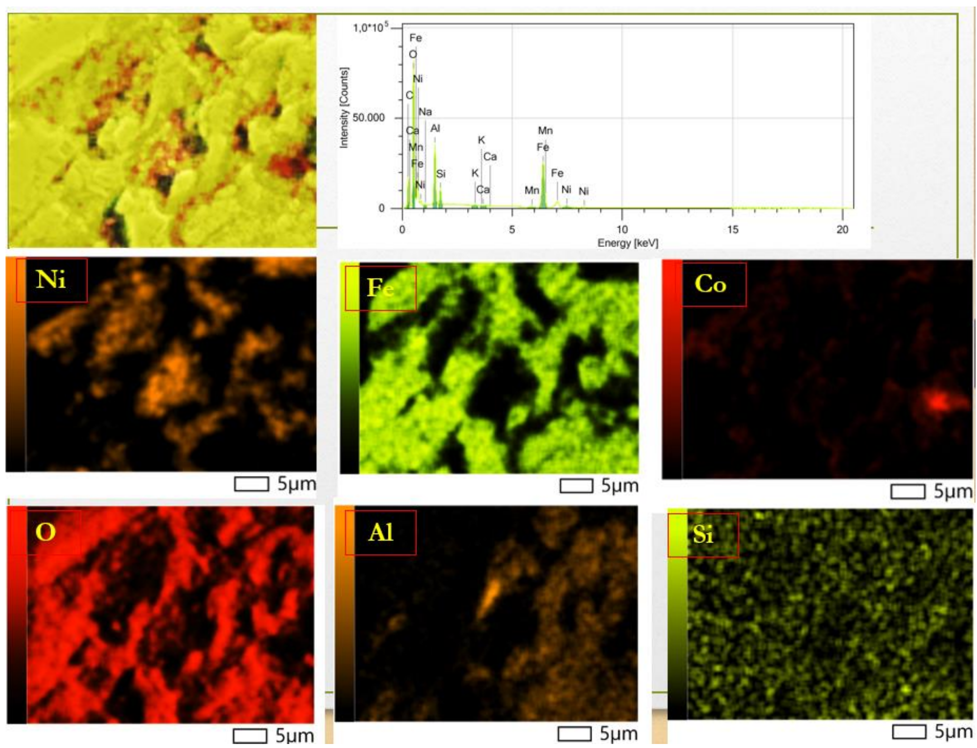


**Figure 8.** EDS mapping on Fe-Ni-Co alloy after 900 $^{O}$C annealing

After the temperature was raised to 1000$^{O}$C, solid solution occurred but contamination with oxygen also increased, the graph shows that oxygen has the highest intensity as shown in Figure.9. It also appears carbon which makes the alloy hard and brittle. This shows that milling for 16 hours and heating temperature of 1000 $^{O}$C in this study is not recommended for alloys 25%Fe-50%Ni-25%Co [19][20]

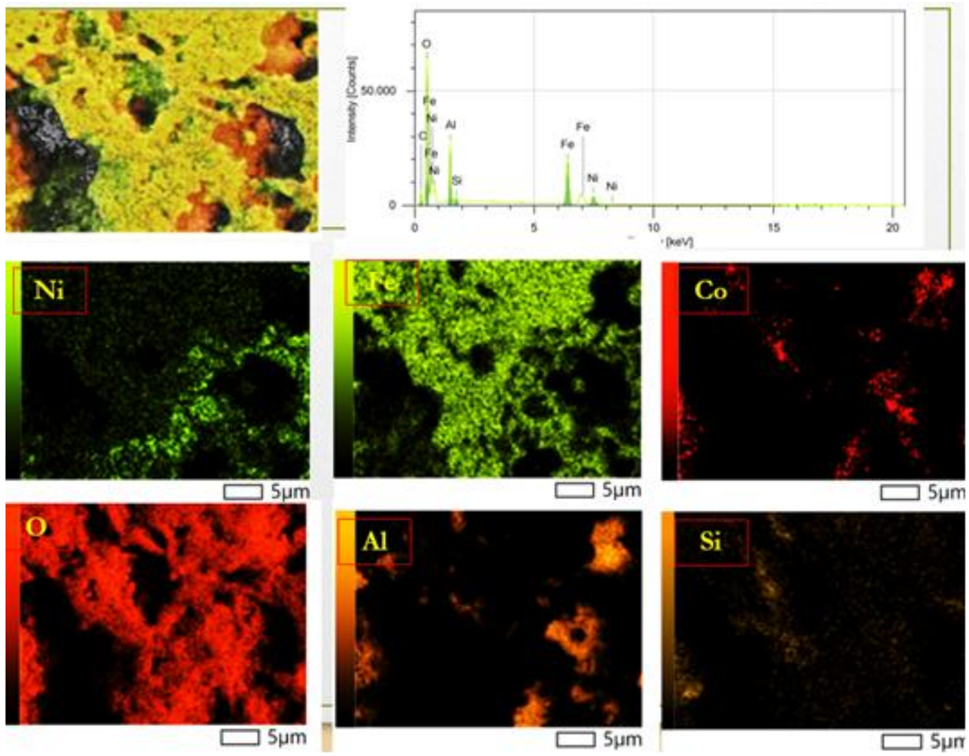


**Figure 9.** EDS mapping on Fe-Ni-Co alloy after 1000 $^{O}$C annealing

### 3.4. Hardness Analysis

The hardness test was carried out using the Vickers hardness test method. The hardness value is obtained by calculating the applied load divided by the average diagonal value of the used diamond pyramid foundation at the position tested. The hardness test was carried out using the micro vickers method with a load of 200 g and a holding time of 5 seconds. The results of the Vickers hardness test (VHN) are shown in Figure 10

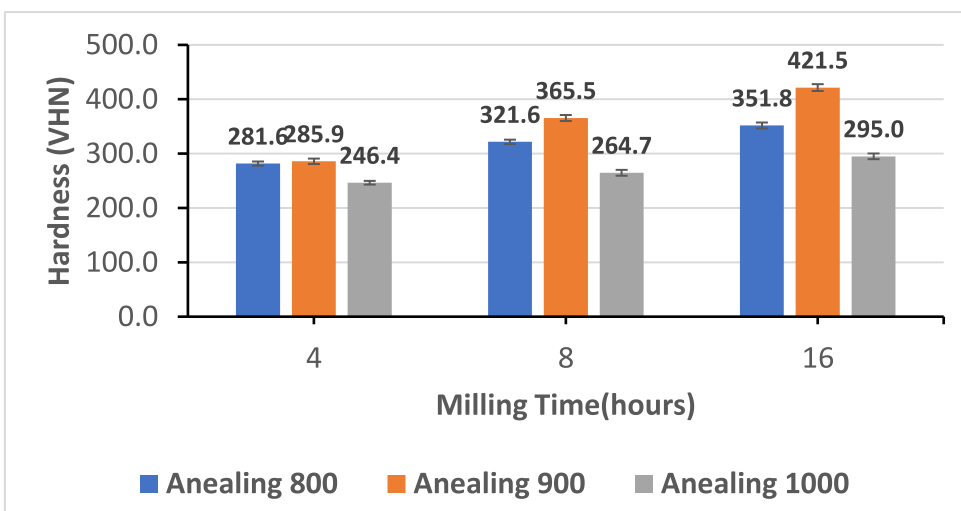


**Figure 10.** Hardness test results with variations in milling time and variations in annealing temperature

Microhardness measurements of Fe-Ni-Co alloys were carried out at different grinding times. The results showed that the hardness value increased with increasing milling time of 4 hours, 8 hours and 16 hours. It can be attributed that the phase distribution of the Fe-Ni-Co alloy is not uniform at the initial stage of milling; However, homogeneous microstructure, grain refinement, and high hardenability can be achieved with long grinding times i.e 16 hours. In addition, the amount of complex Fe-Ni-Co nano-oxide embedded into the specimen inhibited grain coarsening, which also contributed to the increase in hardness. In this study the Fe-Ni-Co alloy was investigated and had the highest microhardness of 421.5 kg/mm2 with 16 hours of grinding time and 900$^{O}$C annealing. It is seen that the Fe-Ni alloy forms a superlattice structure which causes high strength and ductility. In addition, elemental Fe becomes unstable and it tends to form Fe-rich oxides, which can provide dispersion strengthening to improve mechanical properties[21].

## 4. CONCLUSIONS

Fe-Ni-Co alloys were successfully prepared using the mechanical alloying method using a milling time of 4 hours, 8 hours, 16 hours with various annealing processes at 800°C, 900°C and 1000°C. In Fe-Ni-Co powder after 16 hours of milling and annealing at 900°C the microstructure of the Fe-Ni-Co alloy shows a homogeneous distribution of microstructures with fine grain structure and high densification. The Fe-Ni-Co alloy after heating to 900°C showed that a large amount of the oxide appeared as dark particles which were found and have been identified as Fe-rich oxides. This implies that heating to 900°C promotes the rapid reaction of the alloying elements and residual oxygen gas results in oxidation of the material. In addition, the Fe-Ni-Co alloy with a milling time of 16 hours has a higher hardness than the milling time of 4 hours and 8 hours. The results showed that the milling time treatment could increase the hardness effect produced by the high energy ball mill which had a significant increase in hardness.

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