Effect of Milling Time and Ball Diameter on Surface Mechanical Coating (SMC)

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ABSTRACT: Reviewing formation of solid solution through providing powder from the surface of ball and also feasibility of surface mechanical coating (SMC) by using mechanical alloying method and effect of the two parameters, milling time duration and ball diameter on these two matters are items that are being reviewed in this article. In this respect, mechanical alloying process in the presence of copper powder and nickel ball has been used. Parameters which are being reviewed are: 1- milling time (5, 10, 20, 60 and 120 hours) and 2- ball diameter (5 and 9 mm). Various analyses including analysis of X-ray diffraction (XRD), scanning electron microscope (SEM) and electron probe microanalyzer (EPMA) have been used in order to obtain the desired results. The reviews indicate that providing powder from surface of ball increases with the increase of milling time and ball diameter. It was also specified that formation of coating on the surface of ball is possible at the time of mechanical alloying process.

KEYWORDS: mechanical surface coating (smc), milling time, ball diameter, epma analysis

1. INTRODUCTION

In the recent years, mechanical alloying (MA) process has had several applications in creation of new materials. High energy of balls of mill lead to creation of alloys with homogeneous, dense and uniform structures [1]. Mechanical alloying process includes continuous repetition of the phenomena cold welding, mechanical penetration and further breakage of powder particles in a ball mill [2-5]. In fact, particles break in collision with balls and new surfaces are created and next impacts lead to welding of new surfaces with each other [6, 7]. Therefore, MA as a processing method, has the ability to synthesis various equilibrium and non-equilibrium compounds including supersaturated solid solutions, semi-permanent crystals and semi-crystalline compounds, nanostructures and amorphous alloys in the solid state at room temperature [8-12]. Copper is considered as one of the most applicable materials in various industries. While copper creates an alloy with other elements, a homogeneous reinforced structure, with wide application, is created [13-15]. Due to proper

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magnetic behavior, corrosion resistance and also high electrical conductivity, the alloy Ni-Cu is used as coating of small electron parts in industries [16]. Crystal structure of nickel and copper have many similarities with one another and the parameters of their lattice are very close to each other [17-19]. In fact, due to the closeness of atomic radius of these two elements at the time of mechanical alloying process, a bilateral penetration occurs between them, thus, the formation of solid solution of Ni-Cu is possible easily and in a short milling time. But in this article, nickel balls have been used instead of steel balls in the presence of copper powder, in order to measure the possibility of providing powder from the surface of ball. Certainly, this matter will affect condition of attaining solid solution. Things that have been considered in this article are:

1. Review and feasibility of entering nickel powder into copper powder from the surface of nickel ball in order to create solid solution of Ni-Cu;
2. Review and feasibility of creation of coating on the surface of nickel balls at the time of MA process;
3. Review of effect of two parameters, milling time and ball diameter on the above two purposes.

2. TEST METHOD

2.1 Sample Preparation

Mechanical alloying was done in high-energy satellite ball mill with a single chamber (model Fritsch P-6), and in a chamber made of hardened steel with the capacity 125mL. Nickel balls with the purity of 99.95% and 5 and 9 millimeters ball diameter and copper powder with the purity of more than 99.99% and particle size of approximately 200 micrometers were used as charging materials. It shall be noted that in this research, nickel powder has not been used. At the time of milling process, nickel particles are separated from the surface of nickel balls due to the breakage phenomenon and enter copper powder which ultimately leads to formation of solid solution of Ni-Cu. In this research, a weight percentage of stearic acid was added to precursor powder as a controlling factor of process, in order to maintain the balance between cold welding and breakage and also preventing powder particles from sticking to one another [20]. In order to prevent the samples from oxidation, the chamber was charged in the "glove box" device and in the presence of argon atmosphere and it atmosphere was completely protected by O ring [21]. Conditions of milling with 5, 10, 20, 60 and 120 hours duration in the presence of balls with 9 and 5 millimeters ball diameter
and parameter 1: BPR=30 and a speed of milling was considered equal to 300rpm. Conditions and parameters which are reviewed in this article have been shown in Table 1. At the end, after the completion of each stage of milling process, the lid of chamber will be opened inside of the glove box in the presence of argon gas and the remained powders will be extracted in order to do next analyses.

Table 1 Conditions of various samples which are being reviewed

<table>
<thead>
<tr>
<th>Groups</th>
<th>Samples</th>
<th>Time (hr)</th>
<th>Ball diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group a</td>
<td>1</td>
<td>5</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>10</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>20</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>60</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>120</td>
<td>9</td>
</tr>
<tr>
<td>Group b</td>
<td>6</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>10</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>20</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>60</td>
<td>5</td>
</tr>
</tbody>
</table>

2.2. Analysis Equipments

Samples, which have been milled by the X-ray diffraction (XRD) model JEOL (JDX8030), were analyzed by using the CuKα (0.15405 nm = λ) radiation with rapid scanning (8 degrees per minute) between 30-100 angles. And also in order to review and determine the size of particles and morphology of the powder, scanning electron microscopy (SEM) model (JSM-5700) JEOL with voltage of 15 to 20 kV was used and electron probe micro analyzer (EPMA-1720 Shimadzu) was used in order to review distribution of elements in the solid solution.

2.3. Method of Calculations

2.3.1. Williamson - Hall Method

Generally, X-ray diffraction (XRD) peaks are used for studying and analyzing the structure of nanocrystal materials, particularly determining the average of lattice strain and grain size [23, 24]. In order to review these properties, various equations have been provided until now, and the equation of Williamson – Hall is the most important one among them [25].

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The first equation in the right side is associated with the size of grin which is not dependent on diffraction angle and second equation of it shows the available micro-strain in the lattice which depends on the diffraction angle. $\lambda$ the wavelength of the x-ray which has been used (in nm), K is the Scherrer equable that depends on the form of crystallite and index of diffraction surface which is places in the range of 87.0 to 1 and is usually considered 9.0. A is a fixed coefficient and it is dependent on a function which has been used for compliance with empirical data of diffraction. The parameter $d$ is the size of grin (in nm) and $\Theta$ is the Bragg angle (in radians). $\varepsilon$ is the maximum amount of strain which exists in the microstructure of the material and $\beta$ is the broadening that us caused by strain and grin size (in radians), which is calculated based on the Gaussian function according to the following equation [26-29]:

$$\beta^2 = \beta_{obs}^2 - \beta_{inst}^2$$

In this equation, $\beta_{obs}$ is width of the peak in the half of the intensity of the sample's maximum and $\beta_{inst}$ is broadening which is caused by the error of the device.

2.3.2. **Cohn Method**

But for calculating the parameter of lattice with the minimum error, we can use the Cohen method which is the result of solving these two following equations.

$$\sum a \sin^2 \Theta = A \sum a^2 + C \sum a \delta$$
$$\sum \delta \sin^2 \Theta = A \sum \delta^2 + C \sum \delta^2$$

In these two, $a = (h^2+k^2+l^2)$ and $A = \lambda^2/4a_0^2$, $C$ is a fixed number and $\delta=10\sin^2 (2\Theta)$.

3. **RESULTS AND DISCUSSION**

3.1. **Results of the Analysis of X-ray Diffraction XRD**

Figure 1 (a) shows the x-ray diffraction patterns (XRD) of the milled powders through 9 millimeters balls (group a) based on the milling time in two ranges (30 -100 degrees and 41.5 -45.5 degrees). By taking the obtained results from the analysis of x-ray diffraction into consideration, it can be seen that by increasing the milling time from 5 to 20 hours, the intensity of the main peak of the element copper will reduce and it will lead to its broadening. We can say that its reason is relevant to partial dissolution of the element nickel.
in copper and thus, formation of the nickel – copper solid solution. A similar phenomenon is seen in the reference [30]. At the time of 60 hours, the peak which is associated with the solid solution nickel – copper appears, and the intensity of it reduces with the increase of time to 120 hours.

By paying more attention to the obtained results from the XRD analysis, it is seen that at the time of 60 hours, peak is placed in a place between the main peaks of nickel and copper which indicates the formation of the nickel – copper solid solution. Another interesting point is that, by increasing the milling time to approximately 120 hours, it is expected from the associated peak to demonstrate the increase of the amount of percentage of nickel. In other words, the relevant peak is transferred towards larger angles, in contrast, this peak has moved toward smaller angles. Disintegration of various surfaces can be considered as the cause of this matter, because the concentration of Cu is so much more than Ni in the coating layers near intersection of coating / field, which have been formed in the preliminary levels of milling. Therefore, percentage of the element Cu in the powder increases with the entrance of particles of these layers into the powder. Thus, we observe the peak moving towards smaller angles. At the same time we can also observe distinct pea of nickel which itself indicates the separation of some parts of nickel balls in the very long stages of milling.

In Figure 1 (b), the x-ray diffraction patterns which are associated with the samples of group b have been provided. As it can be seen, in sample of 5 hours of the Figure 1 (b), the main peak has moved towards smaller angles, because given the low amount of the energy that has been enforced to powder (both due to the short milling time and small ball diameter), it is not possible for the powder to stick to the surface of ball. Thus, the powder particles which are suspended between balls are exposed to shear forces and face a sever lattice strain. But when a longer time passes, the energy which is required for separating some parts of the surface of nickel ball will be provided and these particles enter copper powder and ultimately, they are solved in each other due to further impacts, and the result of it can be seen as a peak of the nickel – copper solid solution in the 20 and 60 hours samples of milling. Meanwhile, the peaks associated with Cu can even be seen in the 60 hours milling time which can be caused by reduction of energy which is forced on the powder at the time of milling process through balls with smaller ball diameter.
Figure 1 x-ray diffraction patterns in two ranges (30 – 100 degrees and 41.5 and 45.5 degrees) for the milled samples based on time parameter for (a) group a and (b) group b

By considering the graphs of x-ray diffraction and the mentioned equation in the previous section, the amounts of micro-strain, size of grin, distance between crystal surface and parameters of lattice for all of the samples were calculated and presented in Table 2. In this table, for each sample of micro-strain from the slope of the straight line of Williamson – Hall graph and grin size were calculated by using Scherer equation and lattice parameter was calculated by using Cohen equation.
Table 2 obtained specifications from XRD analysis for samples of the groups (a) and (b)

<table>
<thead>
<tr>
<th>Groups</th>
<th>Samples</th>
<th>2θ Cu(111)</th>
<th>Grin size (nm)</th>
<th>d_{111} (Å)</th>
<th>Strain</th>
<th>Lattice parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group a</td>
<td>1</td>
<td>43.30</td>
<td>40</td>
<td>2.088</td>
<td>0.25</td>
<td>3.601</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>43.32</td>
<td>22</td>
<td>2.086</td>
<td>0.25</td>
<td>3.608</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>43.38</td>
<td>16</td>
<td>2.084</td>
<td>0.36</td>
<td>3.660</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>43.90</td>
<td>27</td>
<td>2.061</td>
<td>0.55</td>
<td>3.536</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>43.58</td>
<td>14</td>
<td>2.070</td>
<td>0.64</td>
<td>3.508</td>
</tr>
<tr>
<td>Group b</td>
<td>6</td>
<td>43.02</td>
<td>95</td>
<td>2.101</td>
<td>0.14</td>
<td>3.598</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>43.26</td>
<td>36</td>
<td>2.089</td>
<td>0.16</td>
<td>3.603</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>43.00</td>
<td>56</td>
<td>2.102</td>
<td>0.22</td>
<td>3.604</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>43.44</td>
<td>25</td>
<td>2.081</td>
<td>0.28</td>
<td>3.600</td>
</tr>
</tbody>
</table>

3.1.1. Reviewing Changes of the Grin Size

The size of the grin has been presented in the Figure 2 (a) for the samples of the groups a and b based on the milling time. According to this graph, at the beginning of this matter, size of the crystal significantly reduces. But, gradually with the increase of the milling time, its reduction process slows down and this process is in compliance with the researches of other researchers [9, 31-34]. By considering the Figure 2 (a), we observe that reduction of the size of grin in 20 hours milling time for group a is twice the reduction of the size of grin for group b.

By doing the mechanical alloying process, the lattice strain increases simultaneously with the reduction of the grin size and ultimately, in order to reduce the strain which is enforced to lattice, phenomenon happens again (Figure 2). In other words, reduction of the size of grin increases the energy of the material. Therefore, reduction of the size of grin itself is a restricting factor for reduction of the size of grin more than before [12, 35-37]. In fact, when the size of grin is in nanometer, the formation of solid solution becomes possible. In other words, reduction of the size of grin causes the penetration of the atoms of the guest element inside of the crystal lattice of the host element to occur more easily. On the other hand, increase of the ball diameter increases the energy enforced to the powder and reduces the size of grin more quickly and ultimately makes the penetration of the atoms easier. This matter not only leads to the reduction of activation energy of penetration, but it also enhances directions with a high penetration coefficient and increase of primary penetration leads to the reduction of penetration distances as well [38].

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3.1.2. Review of Changes of Lattice Strain

Figure 2 (b) shows the lattice strain graph based on the milling time for the two groups a and b. As it can be seen, by increasing the milling time, the lattice strain for both groups will increase. Whereas, the increasing process in group a is more than group b, the cause of this matter can be looked for in the intensity of the energy enforced on the powder by balls, in the way that bigger balls have higher impact energy.

In satellite ball mill, rotation of chambers around its own axis and also simultaneous rotation and in the opposite direction of their holder page around the central axis of the device leads to creation of centrifugal force and impact, and as a result of application of these forces to the powder, the mechanical allotting process is done. Therefore, increase of the weight of ball increases the forces applied to the powder and thus, using balls with larger ball diameter applies more energy to the powder. In such way that increase of lattice strain in 60 hours milling time in group (a) is twice of the increase of lattice strain in group (b).

3.1.3. Review of Changes of lattice Parameters

In this article, in order to review the changes of parameters of copper lattice ($a_0$), the Cohen method was used. Figure 3 (a) shows the relationship between copper lattice and time for two different ball diameter. In primary stages of milling with 9 mm balls, the lattice parameter increases and after 10 hours, it reaches the maximum which is 3.660mm. Gradually, with the increase of milling time, the lattice parameter reduces logarithmically, until it ultimately reaches the amount of 3.508 mm for the equable of copper lattice in 120 hours of milling. While, in sample of group b the changes of lattice parameter is not that tangible, and the cause of it can be related to the applied energy being low.

How the lattice parameter in group a increases can be related to expansion of crystal lattice of copper in the early stages of milling processes [39]. In facts, increase of
temperature in the early stages of milling, in which copper is still pure and the particles of nickel have not penetrated it, expands the crystal structure and increases the lattice parameter. Nevertheless, after a while the temperature reaches a fixed rate and the increasing process of the lattice parameter is stopped. But reduction of the constant of cooper lattice after 20 hours of milling has various reasons and different assumptions can be imagined: firstly, on one hand, with continuation of milling processes, the particles of nickel are gradually separated and they enter the copper powder and the dissolution of nickel atoms occurs in the crystal lattice of copper and due to the atomic radius of nickel's being small compared to copper, the lattice parameter slightly decreases. On the other hand, with the increase of intensity of milling, the crystal defects including atomic vacancies, dislocations, grin boundaries and etc. increase, and this itself is considered as a factor for enhancing penetration of nickel and therefore, reduction of the lattice parameter [9, 39]. Secondly, another probability that can be a factor of the reduction of lattice parameter of copper is relevant to the available oxygen in powder at the time of milling process. In such way that, although the entire process of milling has been done under the atmosphere of argon, but the penetration of oxygen during the milling process is unavoidable and the available oxygen in the powder permanently increases with the increase of milling time (Figure 3 (b)) [9]. In preliminary times of milling, while the percentage of oxygen atoms in the atomic lattice of copper is slight, the lattice parameter of copper slowly increases given the probable reasons that were mentioned and then with the increase of the percentage of oxygen, it significantly decreases.

Figure 3 (b) shows the changes between the concentrations of oxygen based on the milling time, according to the obtained results of electron probe microanalysis EPMA. By considering the presence of oxygen in the powder, it is possible for the mechanism of dissolution of oxygen atoms to change after the saturation of crystal lattice of copper and this phenomenon has been expressed by the previous researchers [9]. In fact, it can be deduced that at first a small amount of oxygen atoms is placed in the vacancies of the crystal lattice of copper in between. At the rest of it, after the vacancies between the copper atoms are saturated, an alternative mechanism of oxygen atoms is initiated in the crystal lattice of copper. Due to the tangible difference of oxygen's atomic radius compared to copper and nickel and small penetration distances between atoms at the time of milling and therefore easier penetration of atoms in each other's crystal structure, the lattice parameter of copper strongly reduces. Thus, after 10 hours of milling, it can be expected that due to the dissolution phenomenon and alternation of oxygen atoms in copper lattices and the difference between atomic radiuses of copper and oxygen compared to the atomic radius of copper and nickel, lattice parameter of copper significantly decreases.
3.2. Results of the Scanning Electron Microscope SEM

Figure 4 shows the morphology of the powder remained from the milling processes at various times of the mill by using 9 mm balls. At the start of the milling process, due to the softness of the element copper, powder has found a layer structure. With the increase of the milling time to 10 hours, due to the breakage phenomenon the layer structure breaks and turns to smaller particles and with continuation of the milling process to 20 hours, due to the hard work phenomenon, the size of particles significantly reduces. Nonetheless, a balance has not occurred yet between the cold welds and breakage. When the milling time reaches 60 hours, the formation of Ni-Cu solid solution completes. In this stage, due to the dominance of the cold welding phenomenon on breakage, small particles become large and the size of particles slightly enhances. In fact, morphology of the powders depends on the intermediate and final stages of milling operations [9, 35]. If the milling process continued to 120 hours, given the high energy applied on the powder, particles' breakage occurs again and the size of them tangibly decreases. In other words, too much increase of the energy applied to the powder leads to disintegration of the powder's particles and increase of its free surface.
Figure 4 scanning electron microscope images of the morphology of the mill's powders based on the milling time a. 5 hours, b 10 hours, c. 20 hours, d. 60 hours and e, 120 hours for group a.

In Figure 5 scanning electron microscope images for the sample of group b have been presented. By considering these images, it is seen that at the time of 5 hours, the broadening sheets of powder are created and then at the time of 10 hours, powder is no longer in the sheet mode and it turns to a layered and complex mode which has not changed with the increase of the milling time. While this steady form can only be observed for the 9mm balls at the time of 10 hours (Figure 4).
3.3. Results of Electron Probe Microanalysis EPMA

3.3.1. Review of Elemental Distribution in Powders

In this article, the device of electron probe microanalysis (EPMA) in order to review the way of distribution of copper and nickel elements in one another. The presented imaged in the Figure 6 has been provided through EPMA device and relevant to the distribution of the two elements nickel and copper in the presence of 9mm (group a, images of the left side) and 5mm (group b, images of the right side) balls for 5, 10, 20 and 60 hours of milling times. It is worth noting that in these images the color spectrum is indicative of the changes of the concentration of the relevant element, in such way that from the color blue towards the color red the concentration of the relevant element increases. By considering the images of Figure 6 the following items can be deduced:

Figure 5 scanning electron microscope images of powder based on the milling time a. 5 hours, b 10 hours, c. 20 hours, d. 60 hours and e, 120 hours for group a

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1. With the increase of the milling time, the concentration of the nickel element in copper powder is increasing for both groups of the samples.
2. Increasing process of the element nickel's concentration in the copper powder in samples of groups a is much more that group b.
3. Large and sometimes layered from particles of nickel inside of the copper powder can be seen in various milling times.
4. Distribution of the element nickel inside of the copper powder is uniformed with the increase of the milling time.
5. Start of the formation of solid solution in group a is at 5 hours of milling and for group b it is at 20 hours of milling.

An argument can be considered for each of the above items is:

1. With increase of the milling time, due to the increase of the number of impacts on the balls' surfaces, a larger amount of particles and surface layers of the balls are separated and enter the powder.
2. Since in group a, the ball diameter is larger than group b, the weight of each ball is proportionally more. Therefore, at the time of ball's encounter, it receives higher energy, and as a result more particles and surface layers are separated from the surface of the balls and enter the powder.
3. Presence of these large and layered particles of nickel inside of the copper powder certainly shows that these particles have been separated from the surface of the nickel ball.
4. Due to the continuation of mechanical alloying process, the conditions have been provided for atomic penetration and therefore, the elemental distribution becomes more uniformed.
5. The connection of ball diameter and the applied energy to the powder certainly has a role in this matter.

Ultimately, by taking the images of the Figure 6 into consideration, the following stages can be extracted about the process mechanism:

1- Separation of nickel layers from the ball surface; 
2- Entrance of nickel layers inside of the copper powder; 
3- Nickel layers' becoming fine and turning into micron particles; 
4- Dissolution of them in the crystal structure of copper; 
5- Finally, simultaneous and continuous repetition of this cycle.
3.3.2. Review of Cross Section of Ball’s Transverse Cutting

In order to review and measure the possibility of formation of Ni-Cu coating and determine the chemical compound of the formed coating layer on the surface of nickel balls, EPMA device was used. The results of the obtained linear scan and microscope images from EPMA device have been provided in Figure 7. By considering the obtained results, it can be seen that at the time of 5 hours, the formed coating only contains copper in which the concentration of nickel has also increased by increasing time to about 10 hours in addition to increasing the coating's thickness. By increasing the milling time to approximately 60 hours, concentration of nickel in the coating becomes approximately equal to concentration of copper and coating reaches its maximum thickness (approximately 60 micrometer) and ultimately at the time of 120 hours, the formed coating has been destroyed.
Figure 7 microscope images of the cross section of balls' transverse cutting (images of the left side) and graph of changes of the concentration of elements nickel and copper from surface towards center of the balls (images of the right side) for various milling times a) 5 hours, b) 10 hours, c) 20 hours, c) 60 hours and d) 120 hours. (Relevant to samples of group a)

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The significant point is that in the 60 hours sample (Figure 7c), the changes of the elements copper and nickel's concentration can be divided to three divisions:

1) Location associated with the ball (location III) and in two other locations relevant to coating which are:

2) Location I which contains coating of solid solution with uniform and homogeneous distribution of nickel and copper in one another; and

3) Location II which contains a not uniformed and homogeneous coating of nickel and copper.

4. CONCLUSION

By considering the results of SEM, XRD and EPMA analyses and the presented analyses in the text, the following items can be deducted:

1. Formation of Ni-Cu solid solution through the system of copper powder and nickel ball, which shows the separation of particles of nickel from the balls' surfaces and their entrance inside of the copper powder.

2. Start of the formation of Ni-Cu solid solution after 5 hours of milling for group a and after 20 hours of milling for group b.

3. Delay in the formation of Ni-Cu solid solution, since supplying nickel form the surface of the balls and therefore the completion of formation of solid solution at the time of 60 hours of milling takes lots of time.

4. Increase of dissolution of nickel inside of the crystal lattice of copper with the increase of the milling time.

5. Reduction of grin size at the time of 20 hours of milling for group a, is twice the reduction of grin size for group b.

6. Increase of lattice strain at the time of 60 hours of milling in group a, is twice the increase of lattice strain in group b.

7. Changes of the lattice parameter can be caused by three key factors: 1- increase of crystal defects, 2- increase of the concentration of oxygen dissolved in crystal lattice (in the form of in between and alternation), 3- entrance of the element nickel inside of crystal lattice of copper.

8. Crushing of particles under the influence of ball diameter parameter, in such way that no tangible change can be observed in samples of group b.

10. Destruction of coating at the time of 120 hours shows the lack of sufficient stability of the created coating on the surface of the ball. Thus, it is deducted that the SMC method cannot be considered as a proper method for creating coating.

11. By considering the entrance of nickel particles from the surface of ball to the inside of powder, it can be deducted that the created coating on the surface of the balls is not sufficiently stable. Of course, the stability of coating certainly depends on the material of ball and powder.

12. From accommodation of the results of various analyses (XRD, SEM and EPMA) for each of the samples, it is deducted that the formation of solid solution has occurred for samples with less than 60nm grin size (for group a after 5 hours of milling and for group b after 20 hours of milling).

13. 5 stages of the mechanism of the formation of solid solution in the system of copper powder-nickel ball are: 1- separation of nickel layers from the surface of the ball, 2- entrance of nickel layers inside of the copper powder, 3- crushing and turning into micron particles of nickel layers, 4- their dissolution in the crystal structure of copper and 5- ultimately continuous and simultaneous repetition of this cycle.

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