




Research Article

Optimizing of micro-hardness of nanostructured Cu–Cr solid solution produced by mechanical alloying using ANN and genetic algorithm

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Abstract

In this study, a feed-forward back-propagation artificial neural network (ANN) was designed for the predicting of the micro-hardness of nano-sized Cu–Cr solid solution. The network with 10 and 18 neurons in the first and second hidden layers, respectively, was created based on the 36 extracted data (7 variables and 36 datasets) from a similar study. Then, the genetic algorithm of the effective parameters of mechanical alloying was developed to specify the maximum hardness of Cu–Cr alloys. Consequently, the milling process was performed to validate the created network according to the obtained factors in the genetic algorithm. For this purpose, mechanical alloying was carried out by ball milling of the Cu–Cr powders at 3 and 4.5 weight percentages, respectively. The ball-to-powder weight ratio was kept at 15:1 in the Ar atmosphere and the milling times for Cu-3wt%Cr and Cu-4.5wt%Cr were 48 and 63 h, respectively. Produced powders were studied by scanning electron microscope (SEM) and X-ray diffraction (XRD). Lattice strain, crystallite size, and internal strain were calculated by Rietveld refinement, using the Maud software. Next, the produced powders compressed via a cold press and annealed at 601 °C. The micro-hardness for the Cu-3wt%Cr and Cu-4.5wt%Cr were 228 and 255 Vickers, respectively, while the predicted micro-hardness by the artificial neural network and genetic algorithm were 237 and 243 Vickers, respectively. The root mean square error was 4.25% in the regression of 0.99063 at the proposed sintering temperature. Finally, the result of the sensitivity analysis shows that the milling time, percentage of Cr, and annealing temperature had the highest impact on the micro-hardness of the products.

Keywords Cu–Cr alloys · Artificial neural network · Genetic algorithm · Optimizing of micro hardness · Mechanical alloying

Abbreviations

Parameters	Description
Weight percentage of Cr	A Fraction of the weight of Cr to the total weight
BPR	Ball to powder weight ratio
Milling time	Rotation time of the cup
Vial speed	The cup velocity
Sintering Temperature	Temperature of the heat treatment
Sintering Time	Time of heat treatment
Micro-Hardness	Final hardness of Cu–Cr after the heat treatment

1 Introduction

For optimizing the performance of copper nanocomposites, it is necessary to make a good balance between the electrical and mechanical properties. Indeed, Cu alloys can be strengthened without any reduction in the electrical properties [1, 2]. Increasing the mechanical properties through the formation of a saturated solid solution and the nanometer-scale sediments is a suitable way to enhance the strength, toughness, thermal stability, and creep resistance of copper. Moreover, in this process, the changes in the electrical properties of the Cu alloys

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is reduced significantly [3]. A very good example here is Cu-Be alloys which have attracted much attention by providing a strength of more than 1000 MPa and maintaining the electrical conductivity of copper. But the main drawback of this alloy, which limited its industrial use, is the high cost and toxic nature of beryllium [4]. High electrical and thermal conductivity, good strength, high ductility, and suitable corrosion resistance are the main features of Chromium. Hence, Cu-Cr alloys kept their strength conductivity at elevated temperatures and they have high electrical and thermal conductivity, high corrosion resistance, and good plasticity [5–8]. These noble properties lead to a wide range of applications of Cu-Cr alloys in separators [9], catalysts [10], electrodes [11], etc.

A lot of methods have been used to produce copper alloys including melting and casting processes [12], powder metallurgy [13] severe plastic deformation (SPD) [14], sol-gel [15], and high-energy ball milling [16]. Among these ways, mechanical alloying (MA), due to the simple eco-friendly process and homogenous dispersion of the second phase, has a special place for the production of copper composites. There are a lot of investigations for the fabrication of Cu-Cr alloys by the MA [17–19], but a few of them focused on the prediction and optimizing the micro-hardness of the produced solid solutions.

The milling process of Cu composites has several major parameters which can affect different properties of copper alloys such as the micro-hardness. Exact prediction of the micro-hardness leads to optimize usage of the MA process for synthesis Cu-Cr alloys. Artificial neural network (ANN) is one of the most powerful modeling tools for approaching different datasets based on learning and prediction [20–23]. According to the application, architecture, and reversibility; ANN can be classified into different types. The most important neural networks are Feed-forward, Perceptron, Back-Propagation, Feed-back, etc. [20, 24, 25]. ANN has been widely used in the MA and owing to the different significant parameters in the milling process, it can help to predict desired outputs [26]. On the other hand, the genetic algorithm (GA) is a strong non-linear tool for approaching various parameters [26]; moreover, this is a very common technique using ANN result as the input of GA for estimating the optimized condition [27, 28].

In this study, different percentages of Cr at the various time of milling were used for the synthesis of supersaturated solid solutions of copper by high energy ball-milling. The fabricated nanocomposites were investigated by XRD and SEM analyses and the changes in the crystallite size, internal strain, and the lattice constant at different amounts of the reinforce phases were studied. Subsequently, heat treatment was performed and the micro-hardness of the alloys was measured by the Vickers Micro-hardness test. In order to design the network,

the effective variables in the ball milling of Cu-Cr include weight percentage of Cr, the ball to powder weight ratio, milling time and speed, sintering time and temperature were considered as the inputs; and the micro hardness of Cu-Cr was selected as the output of GA. In this way, the mentioned factors were collected from the report of Akbari et al. [29]. Then, the optimal data for achieving the maximum micro hardness was obtained from the model of the genetic algorithm and the experimental results of the process. Finally, sensitivity analysis based on the designed network was performed to distinguish the most important factors in the process.

2 Experimental

Precursors were pure Cu (99.7%, <6 μm) and Cr (97%, <250 μm). Samples with two compounds of 3 and 4.5 weight percentages of Cr milled in a planetary ball mill (Sepahan84D) at Ar atmosphere and vial speed of 250 and 247 rpm, respectively. The initial amount of powders was 15 g, the balls were 10 and 15 mm in diameter and the ball-to-powder weight ratio was kept at 15:1. The milling time for Cu-3wt%Cr and Cu-4.5wt%Cr were 48 and 63 h, respectively. The main reason for choosing the mentioned parameters is that the highest micro-hardness which was obtained in these conditions by similar studies [29, 30]. To avoid the temperature rising during the milling process, 15 min rest time was used in every 30 min. According to the Suryanarayana curve [31], injected shock power was in the range of 1–2 W/g.

The structural changes in synthesized samples were studied by an X-ray diffractometer (Philips X'Pert, Cu-K α , $\lambda=0.1542$ nm). Rietveld refinement was used to calculate the copper crystallographic parameters including lattice parameter, crystal size, and internal strain. The main feature of the Rietveld method is the ability to compare the obtained experimental XRD patterns with the information of structural models of different materials to calculate the characteristics of the formed phase. Furthermore, MAUD software based on the Rietveld method, which has high accuracy in the calculation of crystallographic properties, was used.

Morphology and size of ball-milled samples were analyzed by a scanning electron microscope (Cam Scan MV2300, 20 kV power, working distance 25 mm). For sintering the mixed powders, the specimens were molded to a diameter of 1 cm and a thickness of 1 mm. Hence, 1.4 g of the synthesized powders were been under pressure a coaxial cold press machine (12 ton). The sintering process was applied in a tube furnace for a half-hour at 601 °C in Ar. The Vickers microscopy test was performed according to the ASTM E 348–89 standard (Duramin20 Strues

Microprocessor) and micro-hardness was carried out with a force of 98.7 mN at 400 magnification for 5 s.

3 Experimental data results

The morphology and the size of particles are shown in Fig. 1. The severe cold working via the long milling time leads to the dissolving of Cr in the Cu lattice. Furthermore, dissolved Cr particles at more volumes are increased, because of more easily crisping and fracturing of copper powders. In general, Cu is more ductile and so fractured more frequently than Cr [30]. Therefore, chromium powder particles are distributed between copper particles. By increasing milling time, chromium particles tend to break and homogeneous distribution of the average particle size is obtained. Generally, mechanical alloying has a high ability for mixing the particles and has a major positive impact on increasing the hardness of the composites [31]. Likewise, homogenous diffusion of Cr in Cu lattice can affect the micro-hardness of the solid solution.

The XRD pattern of the unmilled Cu and Cu-4.5wt%Cr before and after 63 h of ball milling is shown in Fig. 2. The intensity of the main peaks of Cu ((1 1 1), (2 0 0), (2 2 0) and (3 1 1)) decreased. This is due to the reduction of dislocations and strains after the sintering process. As this was reported by Shkodich et al. [32], the change in intensity of Cr peak is not very noticeable, owing to the low chromium content; however, it was very important to note that the creation of Cu-Cr confirmed by XRD patterns.

Table 1 provides information about the lattice constant, crystallite size, and internal strain before and after the heat treatment. The low percentage of errors indicates the accurate calculation of crystallographic parameters. After heat treatment, the crystallite size was increased from 77 nm to 120 nm. Conversely, the lattice constant and micro-strain was decreased from 36.15 nm to 36.11 nm and 0.006 to 0.003, respectively. Crystallite size reduction (under 100 nm) in Cu-Cr with a similar amount of chromium and milling condition is also reported by Akbari et al. [29]. Decreasing Lattice constant and internal strain and increasing crystallite size after the annealing process have a significant impact on the micro-hardness which is reported by similar investigations [29, 33]. During the ball milling of copper composites, powder particles are severely deformed by the impact of the balls which cause an increase in the local temperature, and as a result, atomic diffusion occurs. Furthermore, the density of crystalline defects such as vacancies, dislocations, and stacking faults are greatly developed. Therefore, the work hardening of particles has been performed over time and as the effects of work hardening expanded, the internal strain was grown and the width of the XRD peaks was

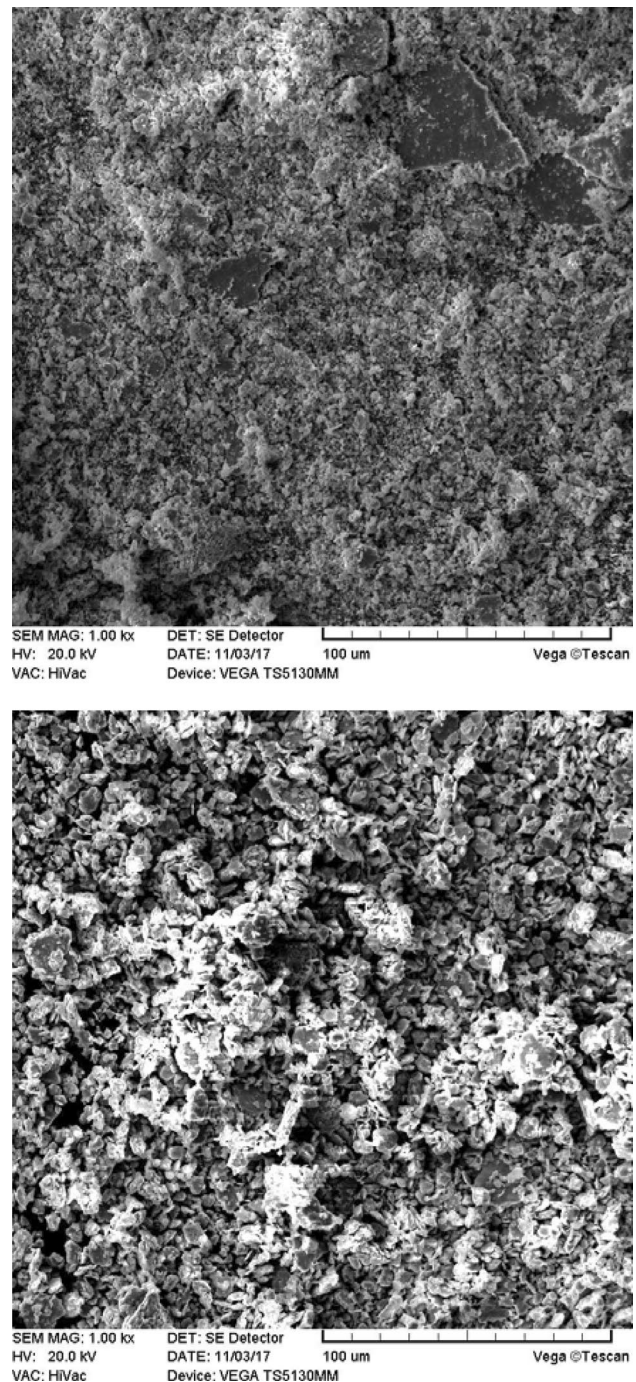


Fig. 1 SEM images of Cu-3wt%Cr and Cu-4.5wt%Cr after the milling process

broadened. The crystal defects are diffusion pathways for the second phase atoms to dissolve in the copper matrix. After annealing and recovery and recrystallization process, the copper lattice parameter is reduced, which is related to the expansion of the reticular parameter of Cu, owing to its bigger atomic radius than chromium. Thereby, during the heat treatment, Cr atoms leave the crystalline structure

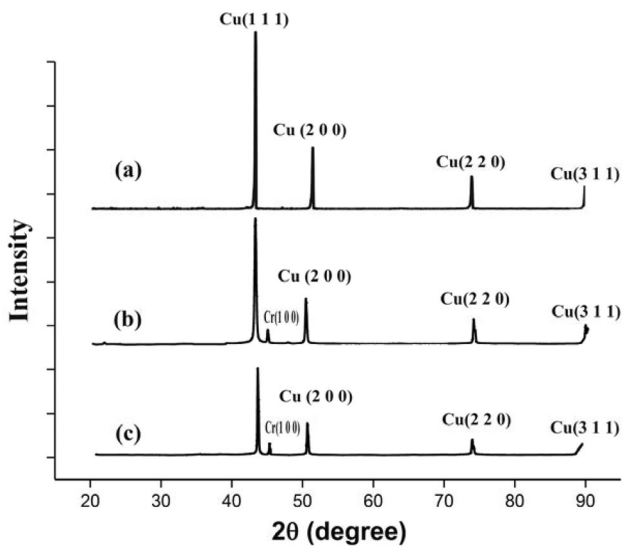


Fig. 2 X-ray diffraction pattern of unmill Cu (a), Cu-4.5wt%Cr before (b), and after the sintering procedure (c)

of the supersaturated alloy and causing a reduction in the reticular parameter of the Cu [34].

In General, the crystallite size is determined by the competition between plastic deformation through the motion of dislocations, and the recovery, and recrystallization [35]. Indeed, recovery and recrystallization increase, and the crystallite size decreases by reduction of the plastic deformation. In this study, owing to the low milling speed (250–247 rpm) and 15 min stop after per cycle (30 min of ball milling), the vial temperature did not increase and so the recovery and recrystallization did not occur, as well as the crystallite size was declined with development plastic deformation. Nevertheless, it is completely acceptable after annealing, adhesion of the particles occurs by surface melting and recovery and recrystallization process; and as a result, the crystallite size grows [31]. Usually, initial powders of different materials are strain-free, but by starting the milling process, there is a rapid increase in the number of dislocations and other crystallographic defects [31, 36]. Gradually, via the formation of dislocations and increasing the crystallite size to a few nanometers, the number of dislocations reaches the saturated extent and under this situation,

new dislocations will not be created by increasing milling time. On the other hand, alloying element atoms also enter into the Cu lattice during this period; so these two phenomena impose strains on the lattice of copper. However, after annealing the strain was reduced due to recovery, recrystallization, and elimination of the dislocations.

4 ANN Modeling procedure

ANN network generally contains interconnected units known as neurons or nodes. Neurons are the smallest computing elements which interconnected to weighted links and they aggregate into layers. These layers affect their input information and can be trained by a process [37]. Indeed, ANN consist of input layers, output layers, and hidden layers and neuron signals transmitted several times from input to the output. The training process of ANN continuous intermittently by changing weights until the network could approach the desired output and reaches to the acceptable error. After training, the network can predict the output of untrained data by using the designed model that was learned at the training step.

The relationship of neurons can be expressed by Eq. (1):

$$x = \sum_{i=1}^p w_i x + b \tag{1}$$

Where the output x produced by the neuron in the layer, p is the number of elements in the layer, $w_i x$ is the weight, and b is the offset or bias.

4.1 Collecting the experimental data

To predict the micro-hardness of Cu-Cr, the experimental data was collected from the report of Akbari et al. [29], and placed in Table 2. The reasons for the selection of this data instead of information of other articles were the same ball milling machine and weather conditions, which can affect the milling process [31] and so the error of the designed ANN model will be reduced.

Table 1 Crystallographic properties (Lattice constant, crystallite size, and internal strain) and Errors of the Rietveld method for Cu-4.5wt%Cr before and after the heat treatment

Cu-4.5wt%Cr	Lattice constant (nm)	Crystallite size	Internal strain	Rw	Rwnb	Rb
Before heat treatment	36.15	77	0.006	4.9868	5.9442	3.8159
After heat treatment	36.11	120	0.003	5.6586	4.6097	4.2066

Table 2 Experimental data sets of Cu-Cr produced via MA (sorted by Milling Time) [29]

No	Weight Percentage of Cr	Milling Time (h)	Sintering Temperature (°C)	Micro Hardness
1	1	4	451	117
2	1	4	602	64
3	1	4	750	44
4	3	4	450	131
5	3	4	601	99
6	3	4	750	31
7	6	4	452	130
8	6	4	602	102
9	6	4	751	38
01	3	12	450	151
11	3	12	599	127
12	3	12	750	45
13	6	12	452	166
14	6	12	602	134
15	6	12	751	64
16	1	12	447	149
17	1	12	599	106
18	1	12	753	31
19	1	48	451	199
20	1	48	601	200
21	1	48	751	155
22	3	48	452	206
23	3	48	601	243
24	3	48	750	186
25	6	48	452	208
26	6	48	604	189
33	6	48	751	176
10	1	96	452	215
11	1	96	602	222
12	1	96	751	169
22	3	96	452	220
23	3	96	601	242
24	3	96	752	191
34	6	96	454	238
35	6	96	602	189
36	6	96	756	184
22	3	96	452	220
23	3	96	601	242
24	3	96	752	191
34	6	96	454	238
35	6	96	602	189
36	6	96	756	184

4.2 ANN architecture

Among the total number of datasets (36), 5 data sets were used for verification of the network, and the rest of them

applied for training the model. Feed-forward back-propagation, which is one of the most suitable ways for the training of the network in ANN, was performed for training the model. This method presents effective solutions for approaching different factors in order to find a solution [38]. The number of neurons in the hidden layers during the training process was determined by trial and error. This network includes an input layer, two hidden layers, and an output layer. There are 10 and 18 neurons in the first and second hidden layers, respectively. The input variables are the percentage of the Cr, milling time, ball-to-powder weight ratio (BPR), milling speed, temperature and time of the heat treatment; and micro-hardness of Cu-Cr considered as the output. Figure 3 provides information about the schematic diagram of the ANN configuration.

The value of the regression calculated by the finite element method and the trained network, as shown in Table 3. According the regression as the criteria, "Logsig" and "Purlin" are the best functions for the hidden and output layers, respectively; which the best-case is the number 10.

The network modeling was written in MATLAB software version R2014a and the Levenberg–Marquardt (LM) algorithm [39] was used to train the network. Furthermore, the log-sigmoid transfer function was applied as an activation function for hidden and output layers. The data sets have been normalized between 0.1 to 0.9 for homogenization according to Eq. (2):

$$N = 0.8 \left(\frac{x - x_{min}}{x_{max} - x_{min}} \right) + 0.1 \quad (2)$$

Where x_{max} and x_{min} are the maximum and minimum values of the parameters, respectively.

The root mean square errors (RMSE) for the designed network was computed by Eq. (3):

$$RMSE = \frac{1}{N} \sum_1^N \left(\frac{|\text{Actual value} - \text{Predicted value}|}{\text{Actual value}} \times 100 \right) \quad (3)$$

Where N is the total number of training patterns.

4.3 ANN results

In order to test the accuracy of the network performance, regression analysis was performed for training and testing data sets. The result of the regression analysis is shown in Fig. 4. According to this graph, the total regression (total regression of test, train, and validation) was 0.99063. For verification of the network, a comparison between experimental and predicted values of data sets was carried out. Regarding Eq. (3), the network error value was calculated by 4.41%.

Fig. 3 Schematic diagram of the artificial neural network architecture with inputs and output

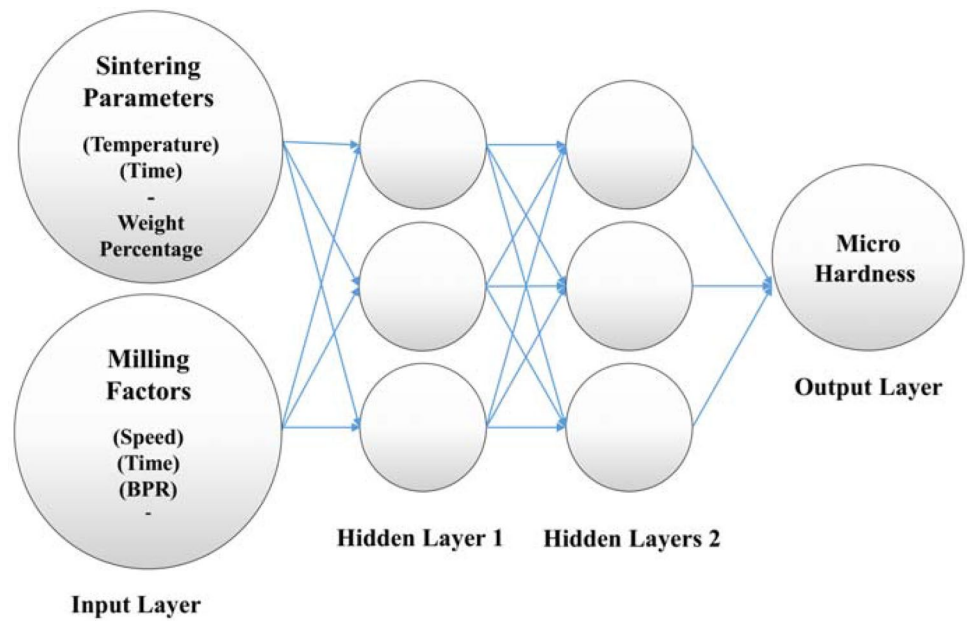
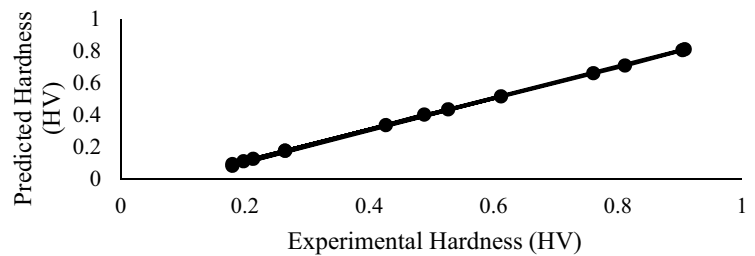


Table 3 The effects of the different Activation function on the MRE (Mean Relative Error)

No	Activation function			Neurons in hidden layers		Mean Relative Error (MRE)
	Layer one	Layer two	Output layer	Layer one	Layer two	
1	Logsig	Logsig	Purlin	3	10	22.47
2	Logsig	Logsig	Purlin	4	1	14.98
3	Logsig	Logsig	Purlin	5	5	66.74
4	Logsig	Logsig	Purlin	12	7	42.19
5	Logsig	Logsig	Purlin	4	20	11.65
6	Logsig	Logsig	Purlin	7	3	57.32
7	Logsig	Logsig	Purlin	15	9	6.75
8	Logsig	Logsig	Purlin	27	30	45.98
9	Logsig	Logsig	Purlin	5	16	8.12
^a 10	Logsig	Logsig	Purlin	18	10	4.41

^aBest ANN structure

Fig. 4 Schematic of regression based on the ANN model for prediction micro-hardness of Cu-Cr



5 Micro-hardness and Genetic Algorithm

Table 4 shows the output data and a comparison between experimental and predicted micro hardness

based on the ANN network. By putting these results in the relation 3, the root mean square error was calculated by 4.25%. According to the genetic algorithm based on the ANN model, the optimum value of each of the effective parameters was measured. 243 and 237 HV

Table 4 Details of micro-hardness tests and proposed GA regarding the ANN network

Alloy	Predicted Micro Hardness (HV)	experimental Micro Hardness (HV)	Percentage error for experimental Micro Hardness	Sintering Temperature (C°)	Milling Seed (rpm)	Alloy
Cu-3wt%Cr	237	228	1.2	601	247	63
Cu-4.5wt%Cr	243	255	2	601	250	48

are the maximum micro hardness of the two optimization modes if mechanical milling is performed with the mentioned parameters.

The micro-hardness of the samples is enhanced by increasing the proportion of reinforcement materials. The enhancement amount of reinforcement material leads to an increase in the lattice parameter and micro-hardness. The alloying element accelerates work hardening, fraction, diffusion, and segmentation of crystallite; thus alloying particles dissolved and the hardness of the matrix grew up. Moreover, by increasing the percentage of the reinforcement materials, the effects of the cold working become more apparent, and as a result, subgrain boundaries and dislocations are created more and more. In Cu-Cr alloys, the reinforcement element increases the temperature of recrystallization and delays the recovery and recrystallization process. Coherent sediments which were formed during the aging of the supersaturated solid solution, cause the cease recrystallization and enhancing the hardness.

As it is clear from Table 4, the designed model with high approximation predicts the results of experimental conditions. Based on this result, it can be expected that the modeled ANN-AG network can optimize other similar results with such high accuracy and reliability. Moreover, regarding the many variables involved in high-energy ball milling, the proposed model reduces the costs and time of the experimental research.

6 Sensitivity analysis

Basically, the influence of each parameter can be determined by sensitivity analysis. This analysis explains which inputs are more important than the other factors. In this test, a step-by-step method was performed on the trained ANN network by changing one input parameter (5, 10, 15, and 20%), and keeping the other parameters constant. Sensitivity analysis was estimated according to Eq. (4):

$$S_i = \frac{1}{N} \sum_1^n \left(\frac{\% \text{Change in output}}{\% \text{Change in input}} \right) \times 100 \quad (4)$$

Where S_i is the sensitivity percentage of each input variable and N is the total number of datasets, i.e., $N = 36$. The changes in the micro-hardness based on RMSE are shown

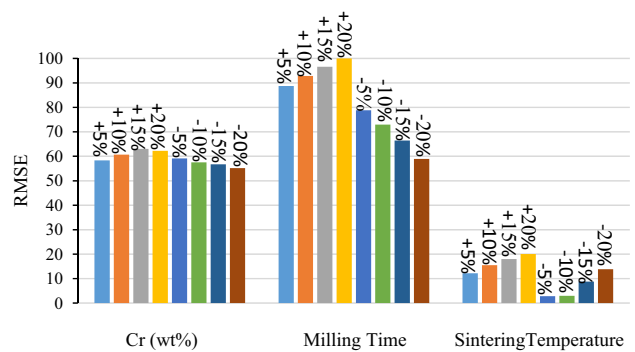


Fig. 5 The effect of the input variables in the micro-hardness of Cu-Cr alloys

in Fig. 5. Milling time and, the weight percentage of Cr and sintering temperature have the highest impact function, respectively. These results match with the XRD analyses (regarding the internal strain, crystal size, and lattice parameter).

Due to the high work hardening of the powders at the ball-milling process, the applied force during compression does not cause significant changes in the micro-hardness [31]. More percentages of alloying elements, the higher density of dislocations, and richer solid solution cause to work hardening of copper and so the micro-hardness of the powders was increased. The rich solid solution in aging will form a high volume fraction of coherent sediments, and crystalline defects provide preferred sites for sedimentation [40]. Increasing milling time leads to enhancement of work hardening and dissolving Cr in the lattice of Cu. Furthermore, more percentage of Chromium rises the micro-hardness of Cu-Cr at high milling time; In fact, high milling time and high volume fraction of Cr intensify each other.

7 Conclusion

In this investigation, first, different Cu-based alloys (Cu-3wt%Cr and Cu-4.5wt%Cr) were produced by high energy ball-milling and after heat treatment of the samples, the micro-hardness and the crystallographic parameters of the alloys were compared and studied. The results of analyses show that the lattice constant and internal strain were

increased, and the crystallite size was decreased by the rise in the proportion of Cr during the milling process. Furthermore, the micro-hardness for the Cu-3wt%Cr and Cu-4.5wt%Cr were calculated 228 and 255 Vickers, respectively. In order to predict the micro-hardness of the alloys, the ANN model with 10 and 18 neurons in the first and second hidden layers was designed, respectively and the produced model was able to predict the micro-hardness of Cu-Cr with an average error of 4.25%. The results of the GA-ANN algorithm show that the proposed model despite the limitation of mechanical alloying such as long time of milling and a large number of milling parameters, is an effective way for the optimization of MA variables and causes the maximum micro-hardness in Cu-Cr composites. In addition, sensitivity analysis represents that the milling time and weight percentage of Cr have the highest impact on the micro-hardness of Cu-Cr alloys. A further study can assess the effect of other parameters like cup temperature or increase the generalization ability of the designed neural network by performing more experiments and obtain a larger statistical population to increase the accuracy of the model and reduce the error of the network.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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