Investigating the Influence of Hardness and Shape Recovery with Sintering Time of Cu-Al-Ni Smart Alloy

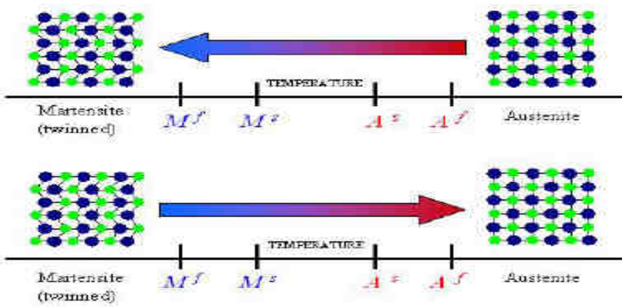
Ahmed Abdulrasool, Hasan Abdulsahib Mezaal

***Abstract-** In this study a Cu-Al-Ni alloy was manufactured by powder metallurgy (PM) method by mixing powder of 83%Cu-13%Al-4%Ni for 6 hrs. after that compacted at 650 mpa and sintered at 850 C for (3,4,5,6,7) hrs. and heat treated to investigate the influence of(hardness and shape recovery) with multiple sintering time. To make sure that manufactured alloy are smart alloy XRD and SEM tests were done for 3 and 7 hrs . The result showed that Martensite layer was formed on surface. The result of hardness and recovery tests showed fluctuation of hardness and shape recovery with sintering time. The effect of sintering time on hardness is apposite on shape recovery. In this research artificial neural network was used to predict the behavior of alloy at sintering time between 3 and 7 hrs.*

***Keywords:** powder metallurgy, hardness, shape recovery ,neural network*

I. INTRODUCTION

A shape memory alloys (SMAs) will recover its original shape after heating the deformed sample. The responsible on this behavior is Martensite transformation. The transformation occur from high temperature parent symmetric austenite phase to low temperature less symmetric Martensite phase[1]. When cool the sample without applying load the material transform from austenite to twinned Martensite. When heating the opposite transformation is occur. Figure 1 shows the transformation without mechanical loading(Ms means Martensite start , Mf means Martensite finish , As means austenite start ,Af means austenite finish) and figure 2 shows the transformation with mechanical loading (shape recovery effect) [2].Cu based alloy is good with high temperature application because of its high temperature application (46 to 146 C) [3].



"Fig."1 Temperature–induced phase transformation of a shape memory alloy without mechanical Loading[2]

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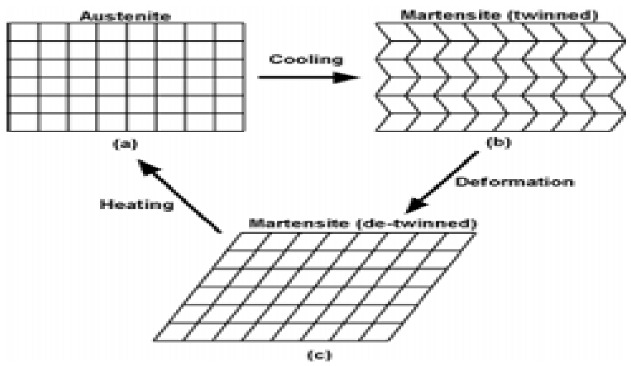
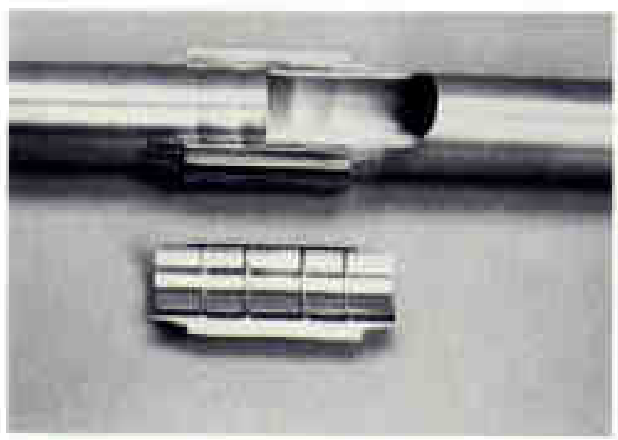


"Fig." 2 the transformation with mechanical loading [4]

A. INDUSTRIAL APPLICATIONS FOR SHAPE MEMORY ALLOYS

There is more than one application that consider Superelasticity the main property of SMAs like eyeglass frame. There is more than one application that consider the Superelasticity at high temperature so may be Cu-Al-Ni alloy is suitable. The recovery application is one of the most common application of smart alloys. The classic application of recovery is Cryofit coupling as shown in figure 3 that hallow cylinder have a smaller inside diameter than tubing system. The hallow cylinder expand to Martensite phase . After installing the cylinder warmed and make a very strong join as transform to Austenite phase. This application is used in hydraulic system in military aircraft. This application may need good hardness to prevent wearing after fastening. There is another applications that consider the actuation property like thermal actuators and safety valves especially fire safety valve as shown in figure 4 this spring may not need high hardness because there is no friction action [5].



"Fig."3 A photograph showing a cut-away section of Cryofit hydraulic fluid coupling[5]

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"Fig."4 industrial fire safety valve[5]

B. Powder metallurgy

It is a fine metal powder blended or mixed. It is then compacted and sintered with sufficient temperature and time to allow contact surface to be bonded in a controllable atmosphere. The compacting is better if it done from two side to increase the homogeneity of density along the sample. The sintering temperature must be 70-80% from melting temperature and may be above melting point for one material if there is more than one material. The lower melting temperature material will flow between the voids of higher melting point materials. The purpose of powder metallurgy is (less machining, high production rates, complex shapes production, controllable properties, and scrap elimination) [6].

C. Artificial neural network

Neural network is a system contains from parallel neurons similar to biological nerves system. The connection between elements determine the network function like in nature. By adjusting the connection between neurons (weights) we can train particular function . Typically, neural networks are adjusted or trained, so that a particular input leads to a specific target (supervised training). The network is adjusted, based on a comparison between the output and the target, until the network output approximates the target. Commonly, many input/target pairs are needed to train a network**[7].**

II. METHODOLOGY

The main goal of this paper is to investigate the effect of sintering time on micro hardness and shape recovery with help of XRD and optical microscope and SEM device . The neural network here used as a tool to predict further values of micro hardness for every 15 minutes from 3 to 7 hrs.

III. EXPERIMENTAL WORK

D. Powder preparation

Cu-Al-Ni was prepared by powder metallurgy method . The powder which has been used in this study has been brought from (Skyspring Nanomaterials, USA) with purity of 99% and an average particle size of 45 micron (-325 mesh). Before mixing the powder was tested by 200x optical microscopy. The first step were all powder mixed with Cu83%-Al13%-Ni4% percentage by horizontal drum mixing with 78 rpm speed for 6 hrs with using 1% percent of acetone (by volume) to prevent particles from separate due to different densities also acetone less the friction between particles. After that the optical microscopy used again to see the homogenization of mixing (the powder was put on white paper and tested).

E. Compacting

Powder mixture were compacted at 650 mega Pascal using cylindrical die of 11 mm diameter. The samples dimension is (11 mm dia. X 5 mm length) and (11mm dia. X 16.5 mm length). The compacting process from two sides to increase the homogeneity of density along the sample. After compacting finish the punch put on hold at 650 Mpa for 2 minutes to prevent the spring back of sample. The optical microscopy with 200x is then used to investigate the microstructure of sample after compacting.

Note: we produced 5 samples, every sample have two specimen with different dimensions as mention above.

F. Sintering

The compacted samples were then sintered in two stages. The first stage at 500 C for one hr. because of Al melting point is 660 C. The second stage is sintering at 850 C for multiple sintering time (3-4-5-6-7) hrs for every sample directly after first stage. then samples were left to cool in furnace at room temperature. The sintering process was done in tube Furness in vacuum atmosphere to prevent oxidization

G. Martensite stabilizing procedure

To transform the austenite samples after sintering to Martensite, two stages were done:-

- Stage one : aging the samples at 800 C for 1 hr. and after that rapidly quenched in iced water
- Stage two: heating the sample to 100 C and hold it at 2 hrs. and left it to cool in furnace .

Stage one and two also done in vacuum atmosphere to prevent oxidization

H. Tests after manufacturing

1) Physical tests

- Optical microscopy : this test were done to see homogeneity of mixing and compacting and sintering and also this test were done to investigate the particles before and after mixing
- XRD: x-ray diffraction tests were done on two samples (3 and 7 hrs. sintered) to cover the range of changes in sintering time. The purpose of mentioned test was for investigate the effect of sintering time on Martensite transformation because of Martensite transformation is the most important step to make sure that the sample is smart alloy.
- SEM: scanning electron microscope was used to obtain clear observation on microstructure to see the Martensite phase .

2) Mechanical tests

- hardness Vickers test: it is important for many application like fasteners
- shape recovery test was done by compact the martensitic samples(Lo) for 4% from its original shape(L1) and then heated to 250 C for 5 minutes and left to cool in air to obtain austenite phase (L2) and then applying the formula below to obtain shape effect.

$$\text{Shape Effect } \% = [(L2-L1)/(L0-L1)] * 100 \qquad (1)$$

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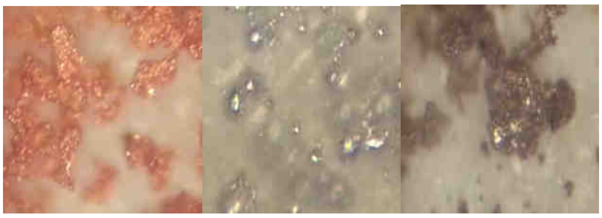
IV. RESULTS AND DISCUSSION

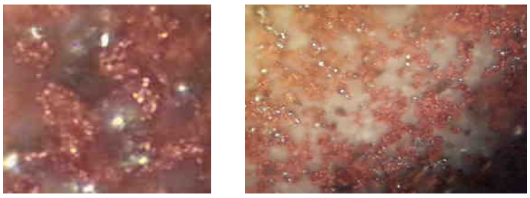
I. The powder testing results

Figure 5 shows the test result of annular single particle of Cu and Ni and spherical particle of Al. this combination make the interference batter than if all particles were spherical. Figure 6-a shows how particles Interfere with each other after mixing process is shown in and we can see also the homogeneity of mixing process as shown in figure 6-b



Cu Al Ni

"Fig"5 fine particles of Cu, Al and Ni

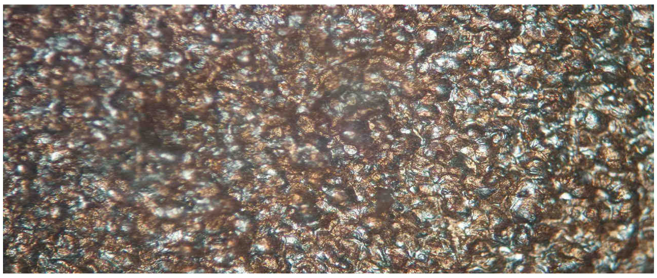


A B

"Fig"6 the particles after mixing

J. The results after compacting

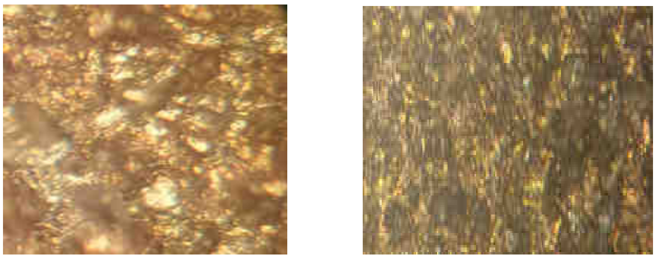
The optic microscopy testing image shows the compacting efficiency and the distribution and homogeneity of alloying element as shown in figure 7. The micro hardness testing result shows drop in its value (65 Vickers because of the usual high porosity and the compacting is not enough to form sufficient bonding force between particles). No need for shape recovery testing because we did not get Martensite phase yet.



"Fig"7 optical microscope with 200X after compacting at 650 MPA

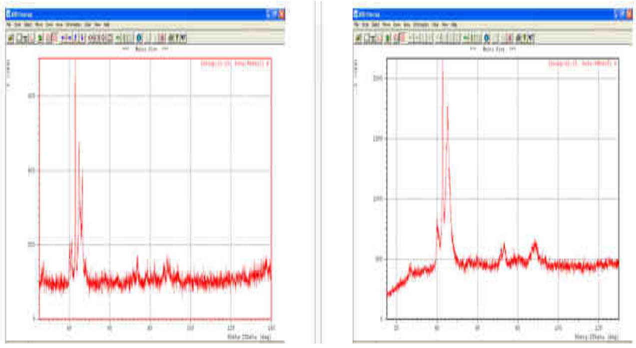
K. Result after completing manufacturing processing at multiple sintering time

1) Optical microscopy tests : the test showed difference in color between sintered samples 3 hrs and 7 hrs samples due to more diffusion between atoms in case of sintering at 7 hrs . Figure 8(A-B) showed this fact.
2) XRD tests : this test showed the peak of Martensite t after quenching and heat treatment at 100 C,. Figure9 (A-B) shows XRD charts.
3) SEM tests : Figure 10 (A-B) shows the Martensite layer that tested by SEM device at sintering time of 3 and 7 hours. The Martensite is more clear in sample sintered at 7 hrs. because of the more sintering time the more diffusion happen so after aging at 800 C and quenching at iced water the Martensite layer was formed in more diffusional alloy than less one
4) Table 1 below shows the hardness tests and shape recovery and figure 11 shows curve between sintering time and hardness at constant temperature (850 C), also figure 12 shows curve between sintering time and shape recovery at constant temperature (850 C) .
5) Figure 13 shows the opposite relationship between shape recovery percentage and hardness. That may because the opposite relationship between hardness and Superelasticity
6) Note : all curves are the direct result from neural network prediction 15 min. sintering time single step input from 3 to 7 hrs.. And plotted by exile



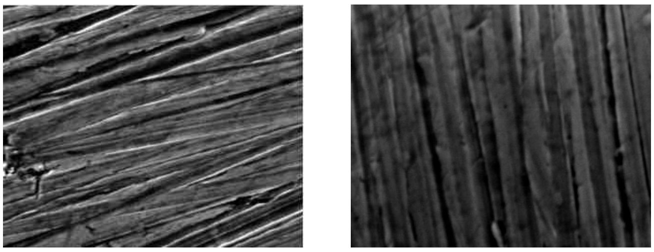
A B

"Fig"8 microscopy test of sintered sample for 3hrs.(A), 7hrs(B).



A B

"Fig" 9 XRD charts shows ALCu3 (Martensite) of two samples: A(3 hrs. Sintered), B (7 hrs. sintered)



A B

"Fig" 10 SEM images shows Martensite layers : A(3 hrs. sintered) , B(7 hrs. sintered)

Table 1 the hardness and shape recovery results

Sintering time	3 hrs. or 180 min.	4 hrs. or 240 min.	5 hrs. or 300 min.	6 hrs. or 360 min.	7 hrs. or 420 min.
Vickers hardness	150	106	85	115	91
Shape recovery %	66	71	80	75	77

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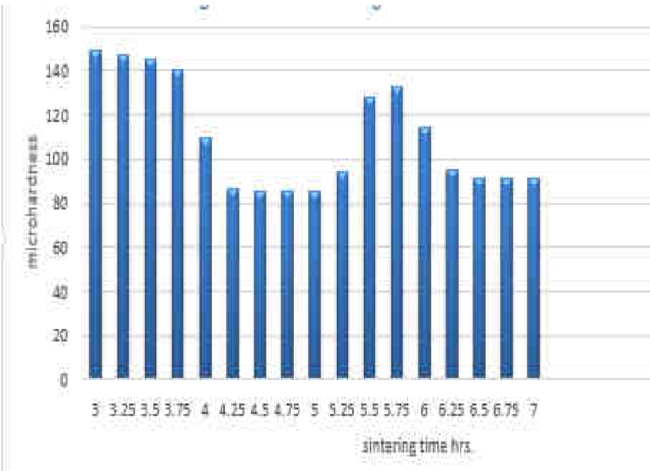
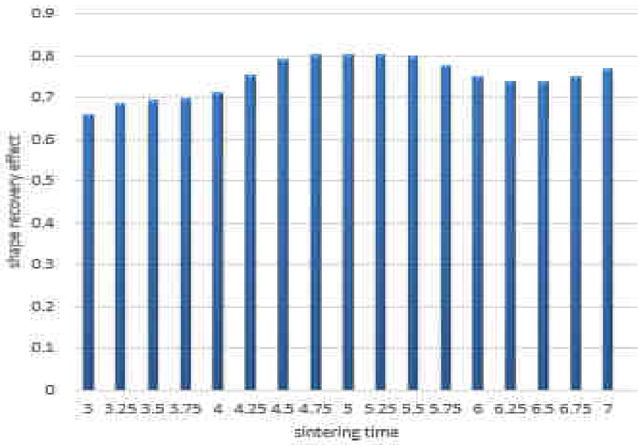
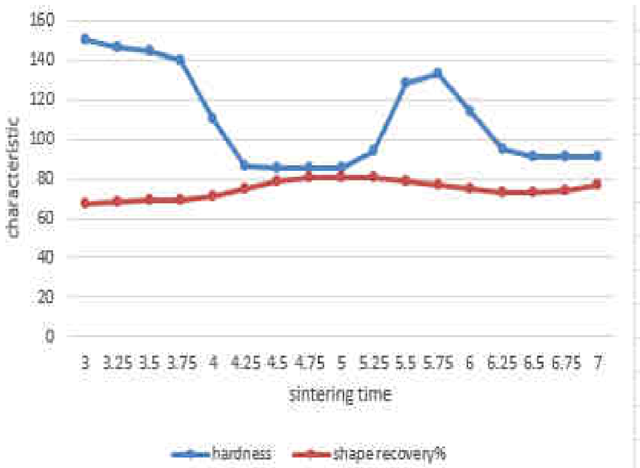


"Fig"11 the relationship between time and Vickers hardness



"Fig"12 the relationship between time and shape recovery

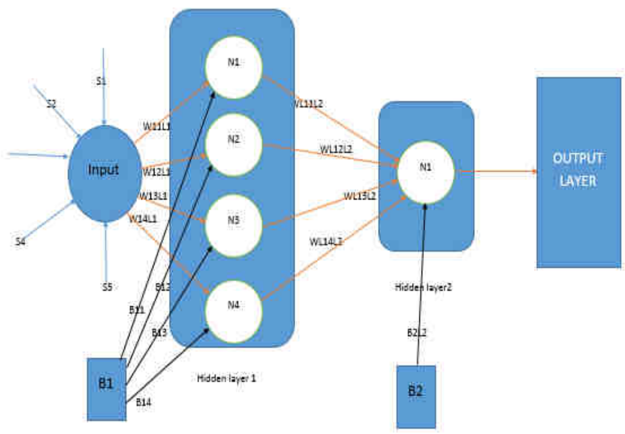


"Fig" 13 the relationship between Vickers hardness and shape recovery with respect to time

V. NEURAL NETWORK PREDICTION

Neural network tools in matlab program are used forward back propagation network to train the input (sintering time) and target (hardness and shape effect results). The performance of training and regression is taken to check the training performance to predict micro hardness and shape recovery at smaller time steps (15 minutes). Network 1 used to train sintering time input and hardness output. The prediction showed high performance at 4 neurons and 2 layers with tan sigmoid transfer function. The other network (network 2) are used at same condition of network above to predict shape recovery behavior at smaller time steps (15 minutes) too. To test the neural network ability to predict a real curve and performance of neural network to predict the behavior of hardness and shape recovery of smart alloy Cu-Al-Ni at multiple sintering time, we done this steps:-

1) Use network 1 and network 2 to predict hardness and shape recovery respectively at every 45 minutes from 3 to 7 hrs. , as shown in table 2, also network 1 and network 2 was used to predict hardness and shape recovery respectively at every 5 minutes from 3 to 7 hrs., as shown in table 3
2) The result from table 2 are used to train another networks (network 3 for hardness, network 4 for shape recovery) but without taking (4,5,6) hrs. as input in consider.
3) Use network 3 and network 4 to predict hardness and recovery respectively at every 1 hr. from 3 to7 hrs. and compare the result with real result that taken from table 1 and compute errors.
4) The result from table 3 are used to train another networks (network 5 for hardness, network 6 for shape recovery) but without taking (4,5,6) hrs. as input in consider.
5) Use same step 3 to network 5 and 6
6) Table 4 shows this comparison and also the weights of every networks and errors between target and network output in network 1 and 2 to check the ability of network prediction. Also table 4 shows the errors between real output and predicting values of network 3,4,5,6. Figure 14 shows the general figure of network with 4 neurons at layer 1 and 1 neuron at layer 2 with full connection between them and with Bias 1 and Bias 2.
7) Excel was used to draw a curves to compare between network 3 prediction output and real hardness output (curve A) and network 5 predicting output and real hardness output (curve B). The curves shows deferent predicting performance due to large input data of network 5 so that neural network work very high quality to predict real data .
8) Curve C and D shows another comparison between network 4 and network 6 for shape recovery. The deference between networks performance have same cause in 7 above. Figure 15 shows curves (A,B) and figure 16 shows curves (C,D) that mentioned above



"Fig"14 general network that used in this study

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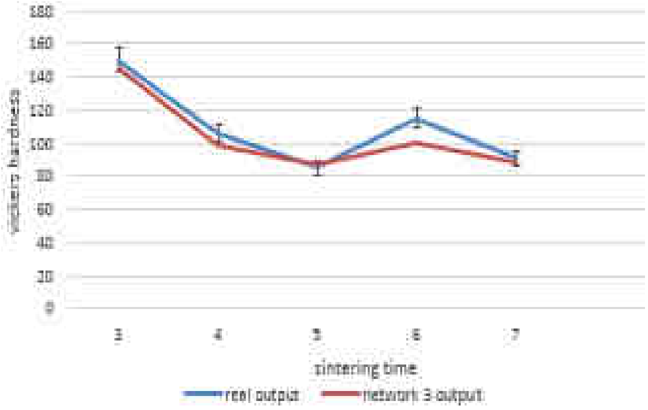
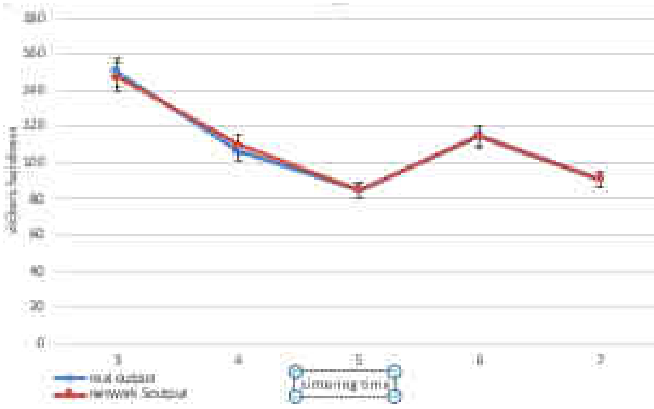
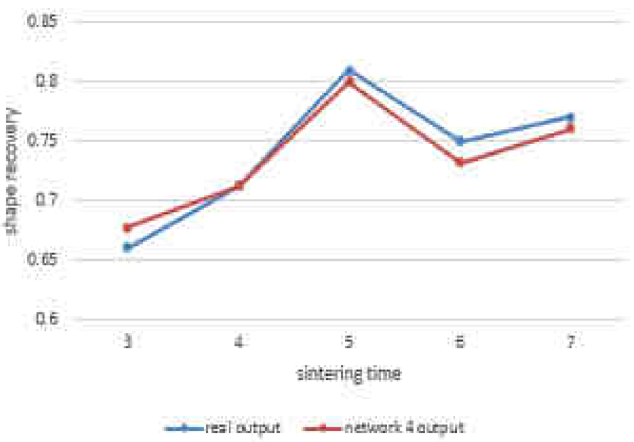


A

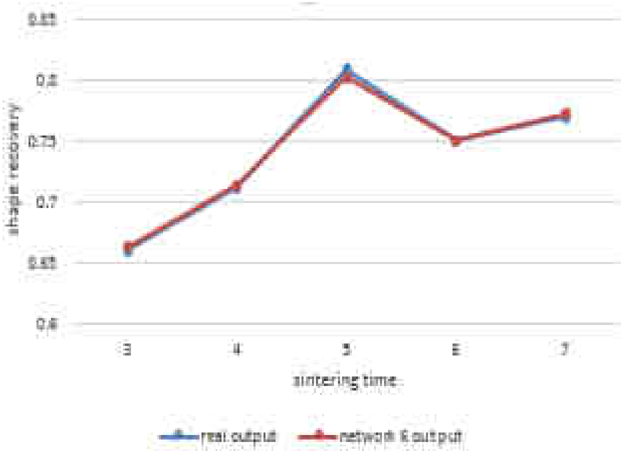


B

“Fig”15 predicting performance of hardness networks



A



B

“Fig”16 predicting performance of shape recovery

Table 2 net.1 and net.2 prediction result for every 45 min. data

Sintering time hrs.	Hardness Vickers	Shape recovery%
3	150	66
3.75	140.2058	69.77
4.5	85.01758	79.22
5.25	93.61496	80.26
6	114.6266	75
6.75	91.14043	74.82
7	91.04047	77

Table 3 net.1 and net.2 predicting data for every 5 min

Sintering time hrs.	Hardness Vickers	Shape recovery
3	150	0.66
3.083333	148.5438	0.68179
3.166663	147.9285	0.68457
3.249993	147.3001	0.687167
3.333323	146.6896	0.689392
3.416653	146.0732	0.691218
3.499983	145.3672	0.692747
3.583313	144.41	0.69417
3.666643	142.8961	0.695733
3.749973	140.2058	0.697758
3.833303	135.0526	0.700683
3.916633	125.2227	0.705138
4	109.7958	0.712
4.08333	95.0273	0.722285
4.16666	87.75177	0.736558
4.24999	85.63403	0.75372
4.33332	85.15226	0.770519
4.41665	85.04467	0.783665
4.49998	85.01758	0.792256
4.58331	85.00964	0.797318
4.66664	85.00746	0.800188
4.74997	85.00821	0.801807
4.8333	85.01314	0.802714
4.91663	85.03113	0.803197
5	85.10684	0.8034
5.08333	85.47697	0.803379
5.16666	87.25202	0.803134
5.24999	93.61496	0.8026
5.33332	106.4823	0.801639
5.41665	119.8671	0.799993
5.49998	128.4797	0.797246
5.58331	132.7306	0.792824
5.66664	134.0924	0.786198
5.74997	133.1881	0.777399
5.8333	129.8177	0.767433
5.91663	123.4677	0.75789
6	114.6266	0.75
6.08333	105.7886	0.744181
6.16666	99.34634	0.740271
6.24999	95.52489	0.737913
6.33332	93.44627	0.736792
6.41665	92.33594	0.736729
6.49998	91.73919	0.737698
6.58331	91.41521	0.739814
6.66664	91.23794	0.743284
6.74997	91.14043	0.748297
6.8333	91.08664	0.754817
6.91663	91.05691	0.76236
7	91.04047	0.77

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Table 4 networks information's

FETURES	Net.1	Net.2	Net.3	Net.4	Net.5	Net.6
W11L1	7.0685	5.609	5.342	5.616	-6.916	13.3813
W12L1	6.1233	5.588	-5.60	5.601	-4.345	13.6828
W13L1	-5.351	-5.60	5.598	-5.52	-1.652	-11.537
W14L1	5.3419	5.639	-5.95	5.761	4.085	12.0424
W15L1	X	X	X	X	X	13.6805
W16L1	X	X	X	X	X	-13.557
W17L1	X	X	X	X	X	-11.519
W18L1	X	X	X	X	X	12.673
W19L1	X	X	X	X	X	13.9567
W110L1	X	X	X	X	X	20.9251
B11	-3.5776	-5.59	-5.857	-5.597	3.479	-14.675
B12	-0.322	-1.89	1.8391	-1.867	-0.449	-11.503
B13	-2.081	-1.86	1.863	-2.119	2.943	5.7038
B14	5.8605	5.560	-5.2494	5.4401	0.607	-4.0917
B15	X	X	X	X	X	-2.6421
B16	X	X	X	X	X	-2.103
B17	X	X	X	X	X	-3.9514
B18	X	X	X	X	X	6.475
B19	X	X	X	X	X	10.3364
B110	X	X	X	X	X	22.0102
WL11L2	-1.0339	0.542	-0.5793	0.7804	1.064	3.0423
WL12L2	3.2298	-0.855	-0.2865	-0.952	-40.33	0.11007
WL13L2	3.4432	-1.142	-0.2646	-1.280	-0.1525	0.2004
WL14L2	-1.4852	0.320	1.3371	0.5289	-40.669	-0.4800
WL15L2	X	X	X	X	X	-0.6867
WL16L2	X	X	X	X	X	-0.7937
WL17L2	X	X	X	X	X	-0.6646
WL18L2	X	X	X	X	X	0.20781
WL19L2	X	X	X	X	X	0.08423
WL110L2	X	X	X	X	X	6.4933
B2L2	1.5915	-0.10	-0.05	-0.269	0.3913	-3.6592
INPUT S1	3	3	X	X	X	X
INPUT S2	4	4	X	X	X	X
INPUT S3	5	5	X	X	X	X
INPUT S4	6	6	X	X	X	X
INPUT S5	7	7	X	X	X	X
OUTPUT1	149.07	0.679	X	X	X	X
OUTPUT2	109.79	0.712	X	X	X	X
OUTPUT3	85.106	0.803	X	X	X	X
OUTPUT4	114.62	0.75	X	X	X	X
OUTPUT5	91.040	0.77	X	X	X	X
TARGET1	150	0.66	X	X	X	X
TARGET2	106	0.712	X	X	X	X
TARGET3	85	0.809	X	X	X	X
TARGET4	115	0.75	X	X	X	X
TARGET5	91	0.77	X	X	X	X
Simulation input 1	X	X	3	3	3	3
Simulation input2	X	X	4	4	4	4
Simulation input 3	X	X	5	5	5	5
Simulation input4	X	X	6	6	6	6
Simulation input5	X	X	7	7	7	7
Prediction1	X	X	145.6	0.677	147.43	0.66314
Prediction2	X	X	98.07	0.712	109.97	0.7139
Prediction3	X	X	87.28	0.799	85.042	0.8028
Prediction4	X	X	100.56	0.731	114.5	0.7505
Prediction5	X	X	88.213	0.760	91.062	0.7729
Real output1	x	X	150	0.66	150	0.66
Real output2	X	X	106	0.712	106	0.712
Real output3	X	X	85	0.809	85	0.8095
Real output4	X	X	115	0.75	115	0.75
Real output5	X	x	91	0.77	91	0.77
ERROR1	-0.9212	0.019	4.3845	0.017	2.5642	0.00314
ERROR2	3.79	0	7.9289	0.000	3.972	0.0019
ERROR3	0.1068	-0.006	2.288	0.009	0.042	0.0067
ERROR4	-0.3734	0	14.44	0.018	0.5	0.0005
ERROR5	0.0405	0	2.787	0.009	0.062	0.0029
MSE	0.5285	0.002	3.855	0.011	1.4280	0.00302

VI. Conclusion

1) The deference in color in figure 8 between 3 hrs. sintered sample and 7 hrs. and the deference between sem images of figure 10 is an evidence that the more sintering time the more diffusion happen between atoms of Cu-Al-Ni particles

2) According to hardness and shape recovery results the relationship between hardness and shape recovery is apposite with respect to sintering time. The fact that shape recovery increase with time because of the more diffusion between atoms the more shape effect ability. The slight decreasing of shape effect after 5 hrs. sintering due to increasing of hardness.

3) Neural network training shows a good predicting ability when there is a constant difference between input stages (network 1 and 2). The prediction ability shows less performance when there is no constant deference between input stages as in network 3 and 4. Even so there is no constant deference between input stages of networks 5 ,6 but the predicting performance is good due to high input data. The point is neural network prediction for one input and 5 input stages is good and we can draw a real curves between output results as increasing input time steps

4) The optimal result for application which needs high shape effect and less hardness is 5 hrs sintering. For application need high hardness 3 hrs sintering is better. For application needs both hardness and shape effect the optimal sintering time is 5.666 as shown in table 5 below

Table 5 the optimal results

Sintering time (hrs.)	Vickers hardness	Shape effect (shape recovery) %	Applications
3	150	66	hardness
5	85.1	80.34	Shape effect
5.666	134.09	78.6	Shape effect and hardness

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