Effect of Nb Addition on Hardness and Wear Resist of Cu-Al-Ni Shape Memory Alloy Fabricated By Powder Metallurgy

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ABSTRACT

Cu-Al-Ni shape memory alloy specimens has been fabricated using powder metallurgy technique with tube furnace and vacuum sintering environment , three range of Nb powder weight percentage (0.3,0.6,0.9)% has been added. Micro hardness and sliding wear resist has been tested followed by X-ray diffraction, scanning electron microscope (SEM) and energy dispersive X-ray spectroscope (EDX) for micro structure observation. The experimental test for the samples has showed that the increase of Nb powder weight percentage in the master alloy has a significant effect on increasing the hardness and decreasing the wear resist therefore it will enhance the mechanical properties for this alloy.

KEYWORDS : Shape Memory Alloy, Cu-Al-Ni, Nb Addition , Hardness, Wear Resist .

تاثير اضافة عنصر النايبيوم Nb على الصلادة ومقاومة البلاء الاحتكاكي لسبيكة Cu-Al-Ni ذات خاصية تذكر الشكل المصنعة بتكنولوجيا المساحيق

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الخلاصة

سبائك ذات قابلية التذكر للشكل Cu-Al-Ni قد تم تصنيعها باستخدام تكنولوجيا المساحيق وباستخدام الخواء كمحيط واقي في اثناء عملية التلبيد ، ثلاثة مستويات للنسب الوزنية لعنصر النايبيوم Nb وهي (0.3 ، 0.6، 0.9) تمت اضافتها الى السبيكة الرئيسية .اختبارالصلادة الدقيقة ومقاومة البلاء الاحتكاكي تمت لجميع العينات والحقت بفحص مطياف الاشعة السينية. وتم استعمال المجهر الالكتروني SEM مع مطياف الاشعة السينية EDX لغرض معاينة البنية المجهرية للعينات . النتائج العملية اظهرت ان زيادة النسبة المئوية الوزنية المضافة من عنصر النايبيوم Nb الى السبيكة الرئيسية له تاثير كبير في زيادة الصلادة الدقيقة وزيادة مقاومة السبيكة للبلاء الاحتكاكي والتي بالتالي تؤدي الى تحسين في الخواص الميكانيكية للسبيكة الرئيسية .

الكلمات الرئيسية : سبيكة ذاكرة للشكل ، نحاس –المنيوم- نيكل ، اضافة عنصر النايبيوم Nb،الصلادة ، مقاومة البلاء الاحتكاكي.

I INTRODUCTION

Shape memory alloys (SMA's) are metals, which exhibit two very unique properties, pseudo-elasticity, and the shape memory effect. Arne Olander first observed these unusual properties in 1938 but not until the 1960's were any serious research advances made in the field of shape memory alloys [1]. The most effective and widely used alloys include NiTi (Nickel - Titanium), CuZnAl, and CuAlNi [2].

The two unique properties described above are made possible through a solid state phase change that is a molecular rearrangement, which occurs in the shape memory alloy. Typically when one thinks of a phase change, a solid to liquid or liquid to gas change is the first idea that comes to mind. A solid state phase change is similar in that a molecular rearrangement is occurring, but the molecules remain closely packed so that the substance remains a solid. In most shape memory alloys, a temperature change of only 10^{o} C is necessary to initiate this phase change. The two phases, which occur in shape memory alloys, are Martensite, and Austenite.
As shown in **Fig.1** The unusual properties mentioned above are being applied to a wide variety of applications in a number of different fields [3].

The shape memory and pseudoelastic characteristics coupled with the bio- compatibility of NiTi make them an attractive candidate for medical applications. The combination of these unique characteristics has led to the development of various applications such as stents, filters, and orthodontic wires.

Also Shape memory alloys have been used in automobiles for applications ranging from impact absorption to sensing and actuation. In addition to the aerospace, transportation and medical industries, there are many other fields and applications that incorporate SMAs.

II. EXPERIMENTAL PROCEDURES

Copper powder with 99.9% purity (-325 mesh), nickel powder with 99.9% purity(-325 mesh) aluminum powder with 99.9%purity (-325mesh) , and Niobium powder with 99.9%purity(-325 mesh) respectively were imported from *SkySpring Nanomaterials, Inc. USA* and used to prepare samples of the master alloy (without addition) with a composition of 83% Cu 13%AL and 4% Ni and for the samples with Nb additives of 0.3,0.6 and 0.9%wieght percentage which was taken from the copper percentage .These powders were weighed accordingly and placed into cylindrical containers which were then mixed in a horizontal barrel mixer as shown in **Fig.2** .

The container was filled with only 50% of powder [4] and 1% of acetone (by volume) was added in order to increase the segregation and prevent the separation of the components (since there is a difference in the densities) .
Alumina balls for assisting the segregation has not been used in order to prevent the milling process and contamination of powder , the speed of rotating drum was set to 75 rpm and the time of mixing was 6 hour .

Samples from powder were prepared in the same die with a cross section of 11 mm in diameter and approximately 17 mm length in average as shown in fig. 3. The samples were pressed at (650) Mpa in a 100 ton Hydraulic computerized press Machine , with a displacement rate of 0.01 inch/min and a holding time of 2 minutes, Between these limits, samples were defect-free and had sufficient green strength for handling.

Fig.3 shows samples from each composition after sintering in an electrical tube furnace supplied with a quartz tube and vacuum equipment

According to the try and error to find the suitable time and sintering procedure and standing on the phase diagram of ternary Cu-Al-Ni , in order to get a fine samples without cracks or defect ,Two stage sintering has been implemented since there is difference in melting point of the alloy components and to prevent the appearance of liquid phase sintering.

The first stage is to sinter the sample at 500ºc for 1 hour and followed by the second stage which is raising the temperature to 850 ºC with soaking time of 5 hour then leaving the sintered sample to cool in furnace. A heating rate of 20ºC/min was maintained for the first stage and 15ºC for the second stage. The vacuum pressure was always allowed to reach 3x10^-6 bar before sintering and during the whole sintering process and cooling. The dual stage vacuum pump is allowed to run for the entire sintering time to suck the harmful gases which will be produced during the diffusing of particles which might effect the sintering efficiency.

After sintering , all sample have been quenched to get ß phase which is AlCu3 (martensite) by heating the sintered sample to 800 ºC and holding it at this temperature for 1 hour then rapidly quenched into iced water . After the quenching process, an ageing heat treatment is implemented to stabilize the ß phase by heating the sample to 100ºc and holding at this temperature for 2 hour [5].

The quenching and ageing process was also implemented in vacuum atmosphere to prevent the oxidation [6].

X- ray diffraction was implemented for the master alloy (without additives) and for the sample with the Nb alloyed element for the three range weight percentage samples.

Scanning electron microscope (SEM) and Energy dispersive X-ray spectroscopies (EDS) have been used to observe the microstructure and the alloying element distribution along the structure.Vickers micro hardness testing with a 1000 gram load (HV 1) has been carried out on all samples with a holding time of 20 second.

More than three values of hardness for each sample have been taken to get the mean value represents hardness. The wear properties of the specimens were investigated using a pin-on-disc sliding wear device, a loading mass of 250 gram and a rotating speed of disc is set to 500 RPM. the pin was located at a radial distance of 100 mm from the rotating axis , for each 5 minute sample has been taken out, cleaned and weighted with 0.0001 g accuracy balance. Six readings have been taken for each case to plot the mass loss against the sliding distance curve.

III. RESULTS AND DISCUSSION

Fig. 6 (a ,b ,c ,d) shows four x-ray charts for master , 0.3Nb ,0.6Nb,0.9Nb additive samples respectively the result peaks was compared with the standard cards with the possible known phases which will be appear.

All samples have shown the martensitic phase after Nb addition and quenching which indicate the existence of the shape effect and no effect of Nb addition to the martensitic phase.

Fig.7 (a,b,c and d) shows SEM pictures of the microstructures of master, 0.3Nb,0.6Nb and 0.9 Nb respectively and showing the alloying element distribution .
The gray particles in **Fig.7(b ,c and d)** represent the Nb particles , the black particles represent the empty pore and the base is the structure .so it is quite obvious that the Nb addition was good distributed in the structures which indicate the successful mixing method and procedure.

As shown in **Fig. 8** and **Fig.9 (a , b)** EDS results for the alloyed sample is showing the existence of Nb in zone 1 which is confirm that this is a Nb particle and shows for zone 2 the existence of the Cu-Al-Ni concentration similar to the specified amount of mixture .

In **Fig. 10** Vickers micro hardness shows the differences between the master alloy and the three weight percentage of Nb additives samples, As it is seen there is a linear increment in HV result with the increasing the weight percentage of Nb which result to alloy enhancement with the addition of Nb particle to the structure, while In **Fig.11** mass loss for master and the alloyed samples has been determined.

Sliding wear resistance results indicate that there is a significant improvement in reducing mass loss

for the samples with additive Ta particles with the master alloy which has addition weight percentage more than 1% will be increasing more weight loss against the sliding wear for this alloy ,This might be due to the strong attractive between the Nb particles and the structure which has been verified by the increment of hardness followed by the enhancement in increasing the sliding wear resistance

V. CONICLUSIONS

1- Using different Nb powder weight percentage will not effect on x-ray diffraction results or the shape memory effect.
2- Increasing Nb weight percentage will:
   (a) Increase hardness.
   (b) Increase sliding wear resistance for the shape memory alloy.
3- Increasing Nb weight percentage than 1% will still Increases the sliding wear resist for this aloy .

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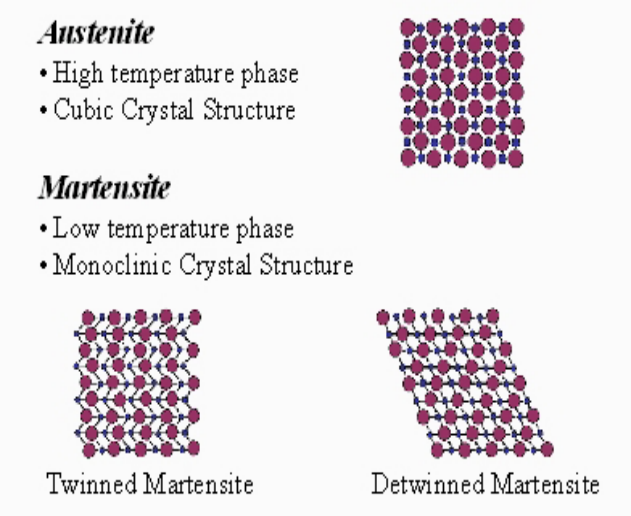


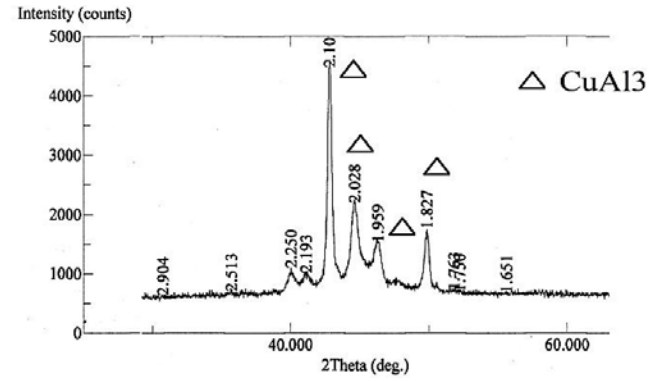
Fig 1: Phases of a shape memory alloy [3]

Fig 2: Horizontal barrel mixer

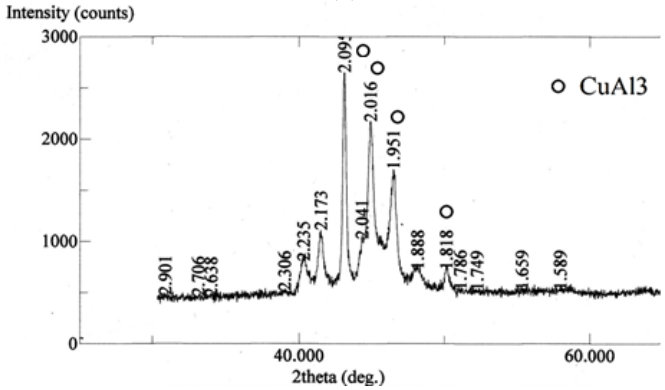
Fig. 3: Sintered samples

Fig. 4: Tube furnace

Fig. 5: Vacuum equipment for sintering process



(a)



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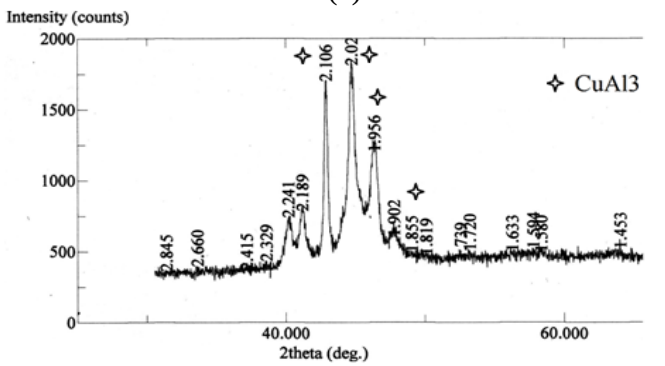

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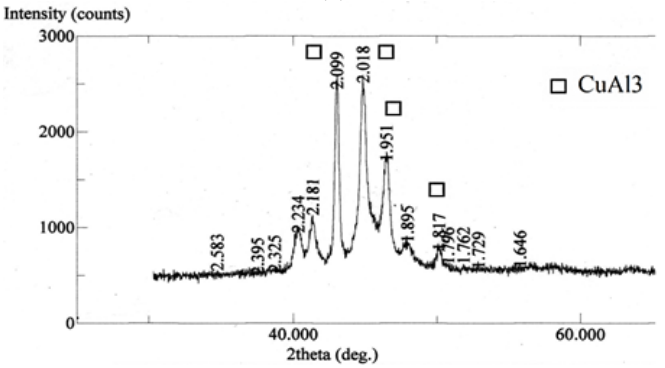


(d)

Fig. 6 (a ,b ,c ,d) X-ray diffractions

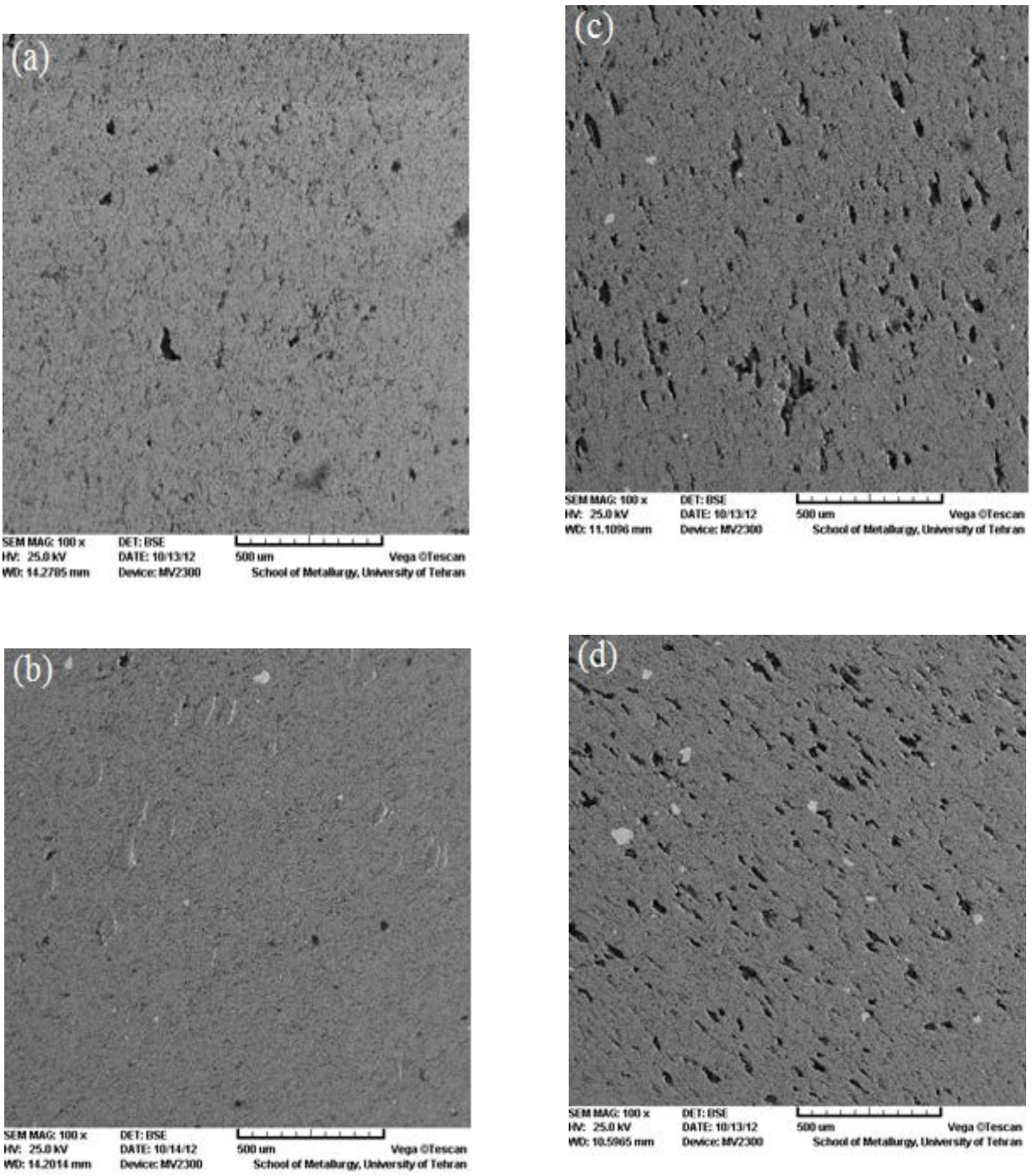


Fig. 7 (a,b,c,d) SEM Pictures with 500X magnification

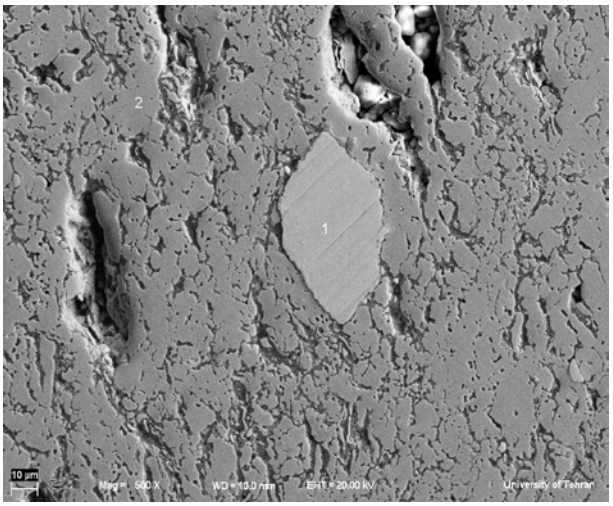
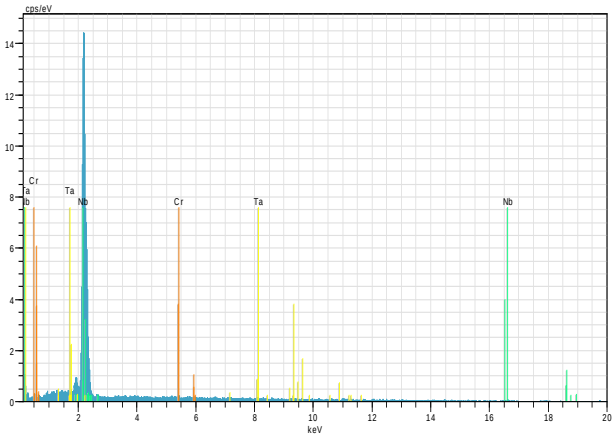


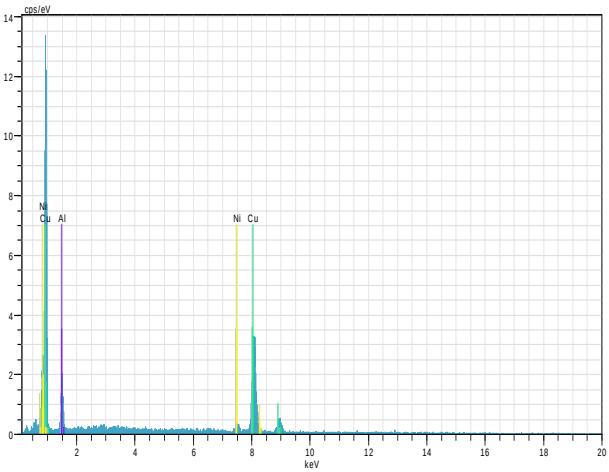
Fig.8 Nb Particle reviled by FESEM



N-1 **Date:10/13/2012 11:35:45 PMHV:20.0kV Puls th.:4.17kcps**

El AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [wt.%]
Nb 41	L-series	122.72	98.64	99.07	4.6
Ta 73	L-series	1.46	1.18	0.61	0.2
Cr 24	K-series	0.23	0.18	0.33	0.1
	Total:	124.41	100.00	100.00	

Fig. 9 (a) EDS results for zone 1



N-2 **Date:10/13/2012 11:36:30 PMHV:20.0kV Puls th.:3.39kcps**

El AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [wt.%]
Cu 29	K-series	71.57	84.99	74.22	2.4
Al 13	K-series	8.78	10.43	21.45	0.6
Ni 28	K-series	3.85	4.58	4.33	0.3
	Total:	84.20	100.00	100.00	

Fig. 9 (b) EDS results for zone 2

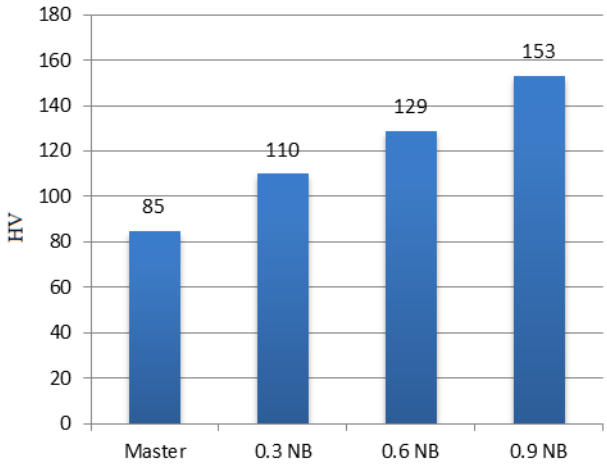


Fig. 10 Vickers micro hardness (HV 1)

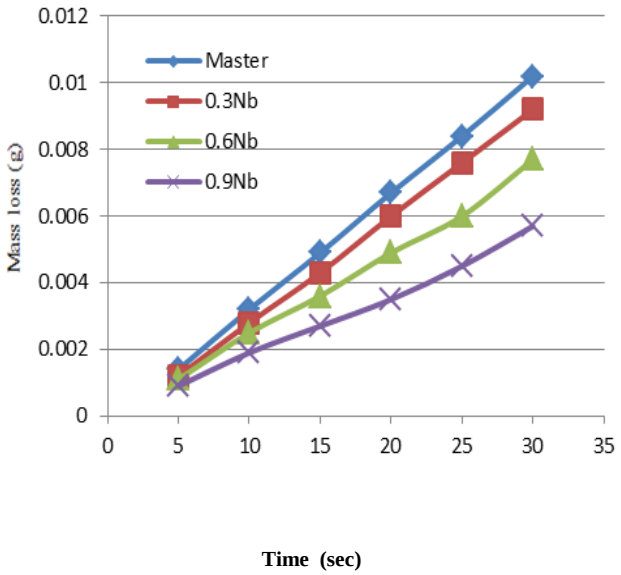


Fig. 11 wear test results