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Synthesis and Characterization of Binary $Ni_{50}Al_{50}$ Alloy Fabricated by Powder Metallurgy Technique

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Abstract

$Ni_{50}Al_{50}$ alloy sample prepared by compacting the powdered alloys at 8 Ton Pressure and Sintered at 750°c for 45 Minutes. The magnetics structural properties and Phase analysis were investigated. X-ray diffraction(XRD), Scanning electron microscopy (SEM), Elements dispersive spectroscopy (EDS) and Vibrating sample magnetometer(VSM) for change in magnetic properties were determined as methods for characterization of produced samples. The results of this alloy show that cubic fcc structure from reflections of (111), (200) and (220). The magnetic results showed that the Saturation Magnetizations (Ms) of the composite alloy substantially decrease. The microhardness values of the alloy was observed that increased with increasing Aluminum addition.

Keywords: Powder Metallurgy, Magnetization, Nickel Aluminum alloys, Microstructure analysis.

1-Introduction

The most common elements are nickel (Ni), Iron (Fe) and Cobalt (Co) or mixtures of them. This materials as 3d transitions exhibit ferromagnetic properties[1].Interest in magnetic behavior has increased recently by mixture of ferromagnetic elements with non-magnetic materials such as aluminum (Al) and Copper (Cu),which naming as a paramagnetic materials [2].The dipoles of Ni as a ferromagnetic materials, tends toward being parallel orientation in an ordered to each other [3]. The AI addition to the Ni is an important parameter to focuse structural and magnetic behavior of an binary Ni- Al and system alloys. Powder metallurgy (PM) processing are widely used to obtain finished materials from pure or mixed metal powders [4].The major steps in PM are powder mixing production, compaction, Sintering and finishing operations such as heat treating [5]. At PM production, of fine powders and even nanocrystalline materials (10-100nm grain size) to obtain enhanced properties is a recent trend and area of interest [6]. In the scope of this study, It is aimed to produce of binary Ni-AI alloys by PM techniques, to realize the structural and magnetic characterization and to find a relationship between structural, morphology and magnetic behaviour in this alloys.

2 - Experimental procedure

Starting materials employed with metal Ni powders of purity 99. 90% with an average effective diameter of ~ 40µm and metal Al of purity 99.95% with average diameter of~ 45 µn. In the first step, Ni and Ai were mixed and alloyed by using mortar and Pestle for 2(hr) until these materials are mixed nicely. After mixing, the powders were added with 5% Poly Vinyl alcohol(PVA) and pressed in 10mm die set under 8 Tons of pressure for 2min .The compacted Samples were annealed at (650,750,850)°C for 45min in Magma furnace with a heating and cooling rate at 5°c/min. The chemical composition and phases. For all specimens were analyzed by using (SEM, model: JEOL,JSM-5410) connected with EDX-SEM.

An X-ray diffraction (XRD)we used investigate the structural and the phases in samples. The patterns were run with Cukα radiation at 40kv and 40mA with scanning speed in 2θ of 4min. The crystallite size of crystalline phases present in the investigated samples was bassed X-ray diffraction line broadening and calculated by using Scherrer equation [7].

$$d=\frac{B\lambda}{\beta\cos\theta}$$

where d is the average crystallite size of phase under investigation, B is the Scherrer constant (0.89), λ is the wave length of X-ray beam used , β is the full width –half maximum (FWHM)of diffraction and θ is the Bragg's angle.

The hardness test of alloys was performed utilizing Vickers hardness machine . Five reading were taken for each specimen of Ni-Al alloy using the load of 0.4kg with 15sec hold time.

The magnetic properties of the investigated samples were measured at room temperature using a vibrating sample magnetometer (VSM,9600-1LDJ,LISA)in a maximum applied field of 15KOp.From the obtained hysteresis loops , the saturation magnetization(Ms),remanence magnetization (Mr)and coerrivity (Hc) were determined.

3- Results and Discussions

Several phases formation reactions was found successfully before and after heat treated at 750°c are shown in Fig.2. The results show that a bigher intensity peaks that were clearly visible for aluminum and nickel phase with metal oxides were identified. New intermetallic compound type(Ni_3Al)phase was observed prospering at angle position (2θ~25.68)with the crystalline size of (30.20nm) was synthesized. Similar results were reported by other researchers [8]. The effect of sintering temperature on porosity at sintering time of 45min on hardness values are shown in Tab.1. It is noticed that as the sintering temperature increased for sample $Ni_{50}Al_{50}$ alloy, the porosity decreased to an minimum point(2.56%) at 750°c with a strongly improved surfaces an hardness value at this sintering point. Above 750°C, a small change start increasing the porosity at 850°c,probably due to the low melting point of aluminum which leads to create many vacancies in the lacttices tends to increase the defects accompanist with porosities[9]. Rahimian et. al.(2009) investigated the influence of size particles sintering time on mechanical properties of powder metallurgy (PM)Al-Al_2O_3 composite and it was reporter that as the size of particle was minimizes, sintering time will increase the hardness values. It was concluded that the best hardness was achieved at 600°c at 45minutes of sintering time [10].

Fig.1(a-b-c)shows the SEM images morphology of binary metals of pure ($Ni_{50}Al_{50}$)alloy at a various sintering temperatures of (650,750,850)°C with time of 45min . Fig.1(a) shows that at 650°C, aside range of distribution with a spherical shape morphology for both elements and started the mselves aggregated slowly .At 750°C sintering Fig.1(b) ,we found that a large rate of diffusion ,without presence of any oxidation during the sintering porccess, it can be clearly seen that the SEM image consist lumps of cluster aggregate spherical shaped particles . Bigger differences microstructure can be seen at 850°C Fig.1(c). We found obviously random orientation between a spherical shape of Nickel and elongated Aluminum particles. Some several Aluminum elongation shape particles preferred a separately movement. No any trace of cluster or lumps aggregated particles was found. It might be that a higher sintering temperature 850°C causes a partial fluidity of aluminum particles or semi molten and then possiply immersed near Nickel particles. From the results shown above ,it can be clearly seen that the SEM image (Fig.1-b) at 750°C is superior microstructure consist lumps of aggregated particles , having the average particles size distribution around 35nm which is comparable to particle size obtained from XRD data. Finally, the results of EDX analysis shows atypical spectrum for $Ni_{50}Al_{50}$ is shown in Fig.1(d) for sintering at 850°C. Both Ni and Al peaks are apparent clearly in the spectrum . It shows also small percentage of Oxygen increased after high sintering only probably due to the plastic deformation and stresses occurs at this temperature[11]. Fig.2 shows the hysteresis loop(Magnetization M versus applied magnetic field H) for selected samples at 300K. Fig.2 (a -b -c) shows the hysteresis loop (Magnetization of versus applied magnetic field H) for $Ni_{50}Al_{50}$ sample at different sintering temperatures. The result show that the sample exhibit soft ferromagnetic behaviour for all sintering degrees [12]. As can be seen in Fig.2, with increase sintering degrees from (650 to 750)°C, Ms was increased gradually from (114emu/g to 127 emu/g) respectively. The results also show at 850°C a declining the Ms Value to. 87 emu/g as shown in Fig.2(c). It is suggested that Ni and Al lattices expanded and plastic deformed in a Ni - Al solid solution during this degree [13].This result actually is identical with SEM. results (Fig.1-c) which observed that intensive thermal energy at 850°C are capable to create many movement and vacancies in the lattices. The results observed also that no significant effect on coercivity (Hc) Values with the change in sintering temperatures. The value of Hc studied by other researchers and reported that coercivity content values depends on changing Nickel and magnetic moment per unit volume only, because Nickel is 3d ferromagnetic transition metal but Aluminum is a Paramagnetic metal, Nonmagnetic behaviour [14]. In this case, in our results show the grain size was not considerably changed between (650 – 750)°C , Kept within the range of(30-40)nm. As the Coercive force (Hc) is a function of magnetocrystalline anisotropy and grain size. For this reason the Hc Parameters have not significantly changed by variation temperatures[15].The Hc Values were obtained about 40 Oe for all cases. The magnetic properties results for the sample $Ni_{50}Al_{50}$ are summarized in Table 3.

4- Conclusions :

The results of this study can be summarized as follows:

- The binary alloy $Ni_{50}Al_{50}$ was prepared by PM method with the suitable high pressure pressing(8Ton) and then sintered at (650,750, 850)°C at sintering time of 45 min.
- Several techniques were used such as, XRD, SEM, EDX an d VSM to analyze the Structural and Magnetic properties in this $Ni_{50}Al_{50}$ alloy. The XRD peaks show increased Peaks significantly with increasing temperatures with a single phase of Fcc structure. No impurity was detected by EDX or XRD indicating that no contamination occurs during.
- SEM images revelaed that typical microstructure with Particles are agglomerated at 750°C,above that at 850°C diffren was structure found that elongated Aluminum particles.
- The Sample exhibits ferromagnetic behavior are at all various temperatures with biggest high magnetic saturations Ms at 750°C about 127emu/gr. The coercive force (Hc) does not show noteable change with 40 Oe and can be classified this alloy as soft magnetic materials.

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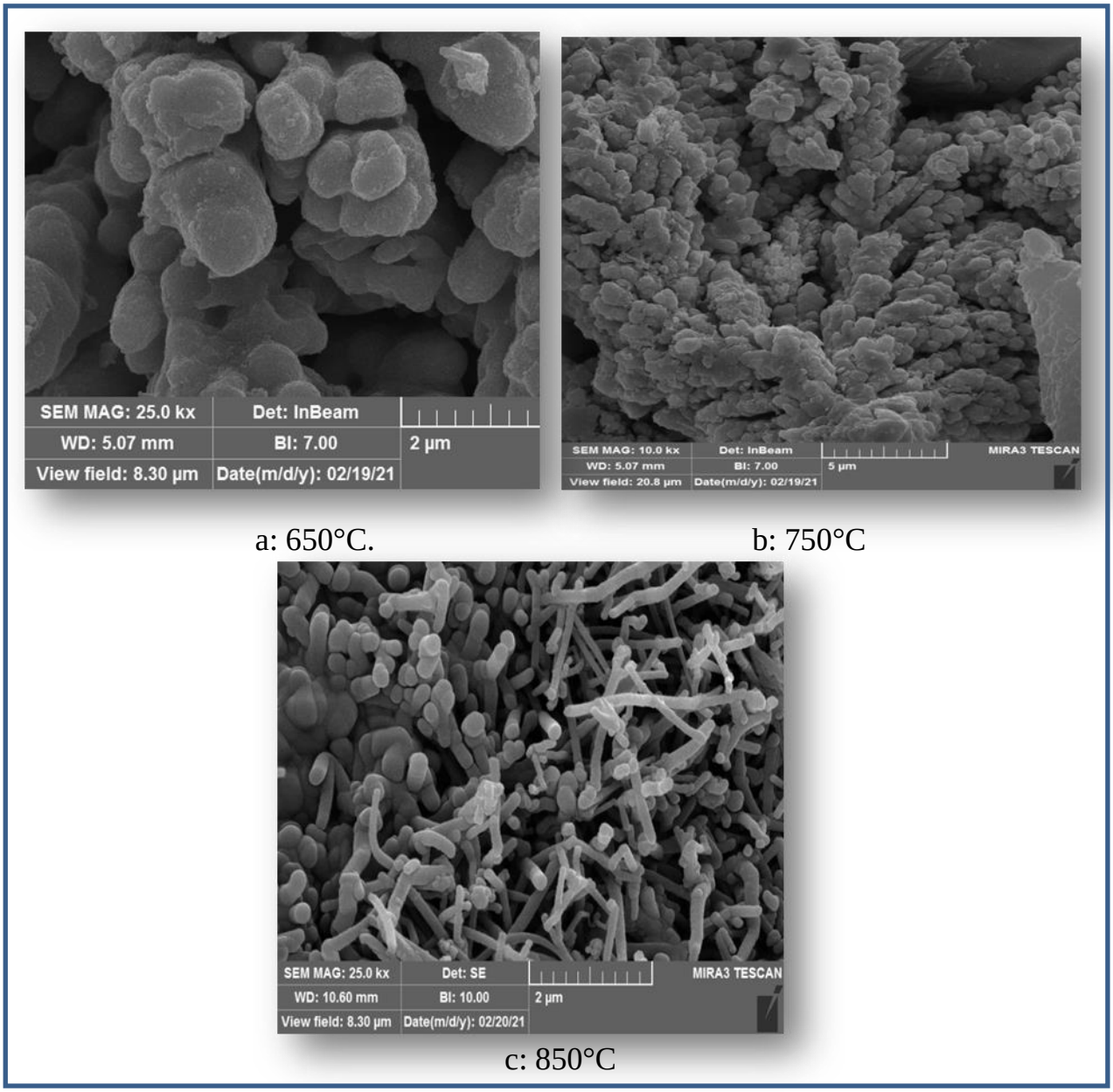

Fig.1 SEM images for $Ni_{50}Al_{50}$ alloy at different sintering temperatures :

a - 750°C
b - 650°C
c: 850°C

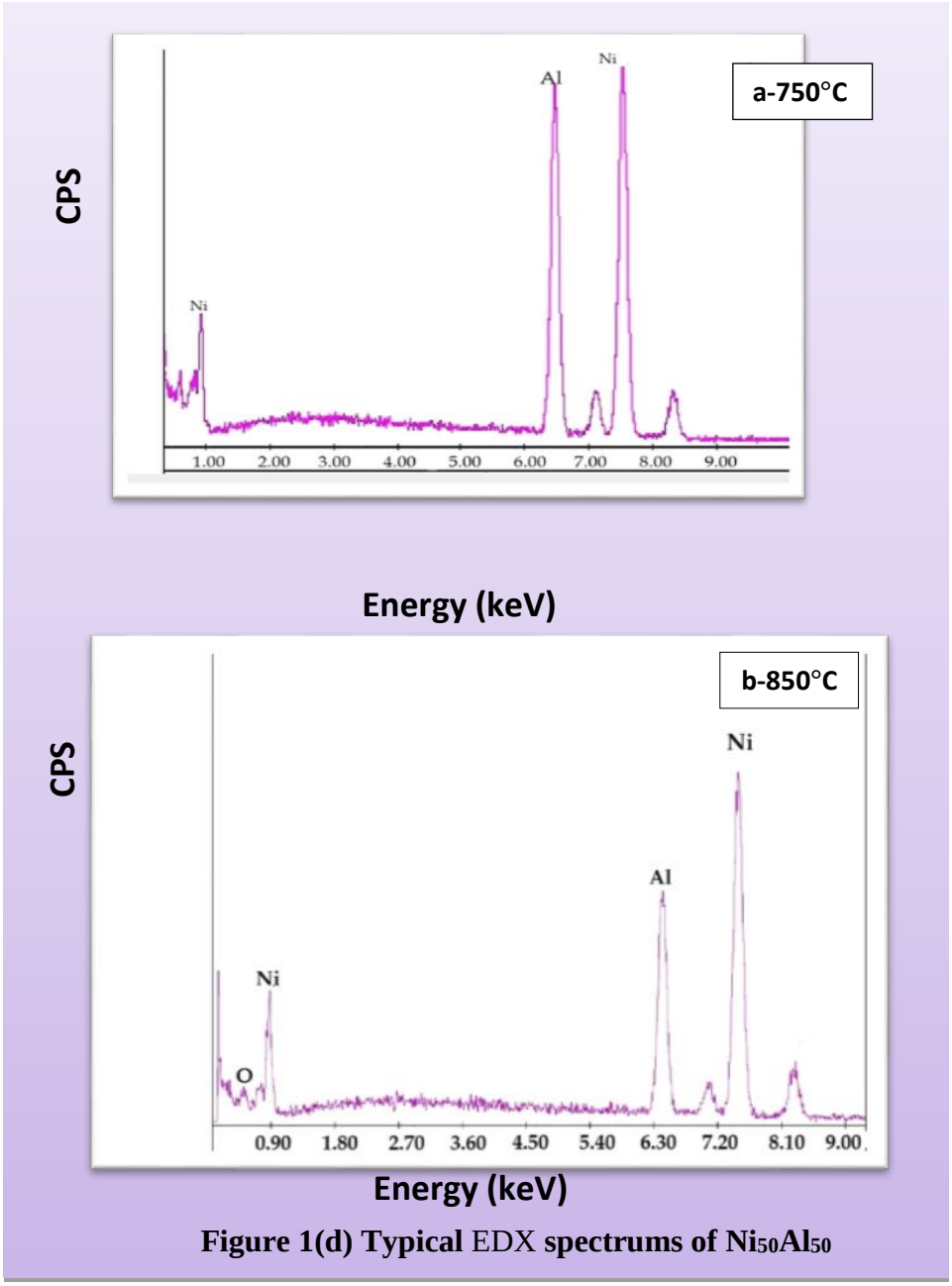

Figure 1(d) Typical EDX **spectrums of $Ni_{50}Al_{50}$**

20(Deg)	FWHM(Deg)	$d_{hkl}(A^{\circ})$	C.S.(nm)	Phase	hkl	Card No.
38.5381	0.2608	2.3342	32.3	Al	(111)	96-901-2430
44.5595	0.2845	2.0318	30.2	Ni	(111)	96-901-3025
51.9083	0.3556	1.7601	24.4	Ni	(200)	96-901-3025
65.1363	0.3319	1.4310	28.4	Al	(220)	96-901-2430
76.4441	0.4979	1.2450	20.3	Ni	(220)	96-901-3025
78.2695	0.4030	1.2205	25.4	Al	(311)	96-901-2430

Tab.1 X-ray diffraction parameters for $Ni_{50}Al_{50}$ alloy at a-RT b-750°C

a-

20(Deg)	FWHM(Deg)	$d_{hkl}(A^{\circ})$	C.S.(nm)	Phase	hkl	Card No.
19.0991	0.2928	4.6431	27.5	Al_2NiO_4	(111)	96-900-5966
25.6757	0.2702	3.4668	30.2	Ni_3 Al	(012)	96-901-3025
31.5991	0.2928	2.8291	28.2	Al_2NiO_4	(220)	96-900-5966
37.4324	0.4054	2.4006	20.7	NiO	(111)	96-101-0382
43.4686	0.3378	2.0802	25.3	NiO	(200)	69-101-0382
44.6847	0.3604	2.0264	23.8	Ni	(111)	96-901-3025

b-

45.40.54	0.3154	1.9959	27.3	Al_2NiO_4	(111)	96-900-5966
49.4369	0.3604	1.8421	24.3	Al_2NiO_4	(331)	96-900-5966
52.0270	0.3828	1.7563	23.1	Ni	(200)	96-901-3025
55.9685	0.4955	1.6416	18.2	Al_2NiO_4	(422)	96-900-5966
63.0856	0.4730	1.4725	19.7	NiO	(220)	96-101-0382
65.6757	0.4054	1.4205	23.3	Ni	(440)	96-900-5966
75.5856	0.5181	1.2570	19.4	NiO	(311)	96-101-0382
76.5090	0.4279	1.2441	23.6	Ni	(220)	96-901-3025
78.0405	0.5405	1.2235	18.9	Al_2NiO_4	(333)	96-900-5966
79.6171	0.5856	1.2032	17.7	NiO	(222)	96-101-0382

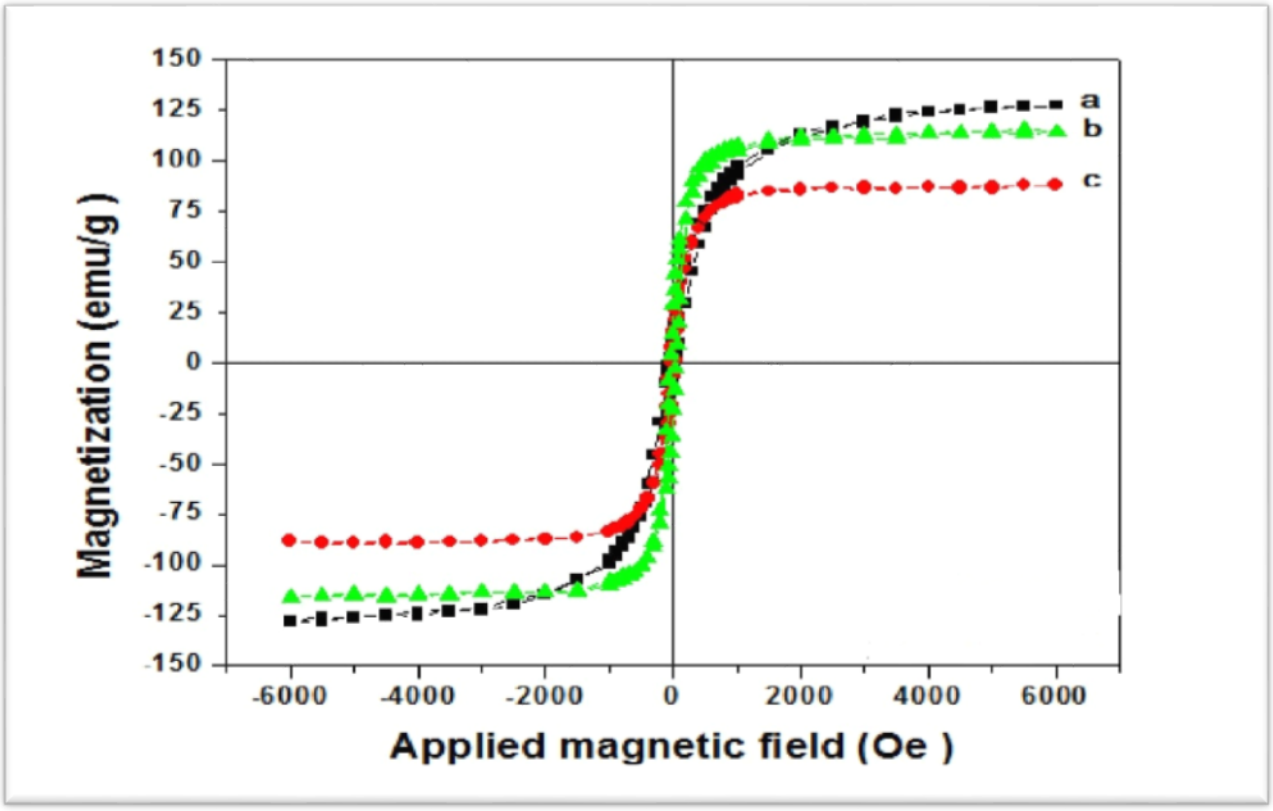

Fig.2(a-b-c): The effect of sintering temperatures on hysteresis loops of the $Ni_{50}Al_{50}$ At a-750°C, b-650°C and c-850°C

Tab.(2): Vickers hardness (Hv) results for $Ni_{50}Al_{50}$ alloy at several sintering temperatures with porosity values

Sintering Temperature °c	T1	T2	T3	T4	T5	Av Hv	porosity %
650.00	205.70	204.33	205.55	207.11	205.31	205.60	6.55
750.00	233.90	234.71	235.40	235.20	234.70	234.78	2.50
850.00	184.50	180.11	170.90	182.80	181.77	180.02	8.80

Tab.(3): The Magnetic and structure properties results for the $Ni_{50}Al_{50}$ alloy at several sintering temperature for 45 min.

Sintering Temperature °C	Coercivity (Hc)Oe	Magnetic Saturation (Ms)emu/gr	Phase Structure	Grain Size (nm)	Magnetic Strucure
650	40	114	Fcc	30-40	Ferromagnetic
750	40	127	Fcc	30-40	Ferromagnetic
850	40	87	Fcc	30-40	Ferromagnetic