Thermal and Structure properties in Ferromagnetic INVAR Alloys Prepared by Powder Metallurgy Processes .

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Abstract

In order to understand the INVAR effect in 3D-transition metal alloys . the $Fe_{1-x}Ni_x$ alloys with x= 0.35,0.50,0.70 have been chosen .

Experiments have been made on thermal and structural properties to study the physical origin of INVAR characteristics such as : thermal expansion , phase transition, and curie temperature Tc which are closely related to its ferromagnetism .

$Fe_{0.65}Ni_{0.35}$ shows an anomalously small thermal expansion coefficient below the curie temperature with (FCC) structure . This behavior has not been found in other combination .

The structural results show that the (BCC) phase occurs above 50% Ni and does not effect the INVAR behavior. The results are discussed with the relation between thermal and structural properties with respect to the magnetic transformation .

Introduction

Powder metallurgy is used in the production of soft magnetic materials for direct current applications and for permanent magnets .In many of these applications , parts are produced by powder metallurgy, because this method permits production to final shape with a minimum subsequent machining and grinding , while at the same time achieving desirable magnetic properties . Nickel is a non –carbide –forming element , which is soluble in iron in all proportions . Nickel helps to prevent excess grain growth at high temperatures and enables fine grain steels to be produced more easily . It tends to stabilize austenite and thus lowers the critical temperature . This makes the heat treatment a little less severe .

Nickel may be present in the steel up to 50 percent . In the range of 2 to 5 percent , nickel contributes great strength and hardness with high elastic limit, good ductility , and good resistance to corrosion and decrease machinability . In the range of 30 to 40 percent nickel lowers the coefficient of thermal expansion , and in the range of 50 percent and above, it increases magnetic permeability .Large amounts give resistance to oxidation at high temperature .

INVAR has practically zero thermal expansion coefficients , which is quite useful for engineering applications . The thermal expansion is a normally associated with the onset of ferromagnetism in certain , notably ($Fe_{1-x}N_x$) with x~0.35 is known as the INVAR effect .

Many experiment and theoretical studies of INVAR alloys have been under taken in recent years using ultrasonic [1] ,neutron secattering[2] and other techniques to elucidate the microscopic origin of the effect , but as yet no clear understanding has been reached[3] . Nevertheless in all models of the INVAR effect it is recognized that the relationship between magnetic and atomic volume or interatomic distance plays a critical role[4] .

The Fe-Ni system is also is of fundamental important from the structure point of view since Ni has a (FCC) structure and Fe is (BCC) .
As an example of ferromagnetism, the ($Fe_{1-x}Ni_x$) alloy system was chosen with (x = 0.35, 0.50, 0.70)

Experimental techniques

Pure Fe and Ni powder supplied by (BDH Ltd.) was used to prepare the alloy as a cylindrical sample of 10mm diameter and 30 mm height . These percentages of Ni are added to Fe and mixed will ball mills for 24 hr. The powder uniaxally pressed at 1000 (kgf) . Sintering was carried out at temperature of 1350°C for 2 hrs of 10 ° C/ min heating rate .

The metallic ratios of the alloys are determined by atomic absorption spectroscopy and energy dispersive spectrometer techniques .

The results of these measurements indicates no evidence of any impurities .The samples for X-ray, DTA were obtained by crushing the alloys in ball mills down to 300 mesh grains .The structure of the ($Fe_{1-x}Ni_x$) alloys (0.35,0.50 , 0.70) were initially investigated using X-ray diffraction at room temperature with a (Philips diffractometer type PW 1877). To obtain lattice parameter more accurately , the Nelson-Riley[5] extrapolation is used .

The specific heat measurements at high temperatures obtained from the differential thermal analysis (DTA type Netzsch 409) where a 1100 C° furnace was used with a heating rate of 10 C°/min . The weights of precipitated samples used were about 500mg . The specific heat determined from the peak area in DTA ,which is depends on the mass (m) and the heat reaction or the enthalpy change (ΔH) ; then using the equation :[10]

$$C_p = [(\Delta H/dT)] .(1/m)$$

Where , dT is the difference in temperature between a sample (S) and an inter reference (R).

A cylindrical specimens ~3 cm in length was used for thermal expansion measurements ranged from room temperature to 900 °C and this was connected to DTA equipment .The relative accuracy for thermal expansion was up to 4% .

Results and Discussion

The aim of this work is studying the new preparing method of ferromagnetic alloys for the binary system Fe- Ni with three ratios by using powder metallurgy method .Structural and Thermal properties have been measured for all samples . A lloys which exhibit INVAR behavior have a small Thermal expansion coefficient below the curie temperature. For alloys in the (Fe_{1-x} Ni_x) system (x= 0.35, 0.50, 0.70) the composition ($Fe_{0.65}$ $Ni_{0.35}$) shows more INVAR effect than other alloys as shown in fig (1) . Both alloys ($Fe_{0.50}$ $Ni_{0.50}$) and ($Fe_{0.65}$ $Ni_{0.35}$) have an (FCC , gamma phase) structure. But the system ($Fe_{0.30}$ $Ni_{0.70}$) has (BCC/ alpha phase) Structure where is no INVAR behavior m The thermal expansion coefficient versus temperature curves are shown in fig. (1).Table (1) Shows the results relevant properties of the three samples .

The reason of low thermal expansion of the system ($Fe_{0.65}$ $Ni_{0.35}$) below Tc (528 K°) due to the interaction between the lattice vibration and magnetic degrees of freedom . However, the above curie temperature the alloy expands normally . This results are identical with the spontaneous magnetization of this alloy which shows anomalies such as deviation of the magnetic moment curve below curie temperature[6] .

The specific heat results for (Fe_{1-x} Ni_x) alloys are shown in fig. (2) which reveal an anomalous temperature dependence .At ($Fe_{0.50}$ $Ni_{0.50}$), C_p- curve is characterized by two distinct maxima : one about 810 k and the second reveals an anomalous maximum at about 540°k . The position of this anomalous maxima is only slightly concentration depended where is a strong decrease of the curie temperature as well as of the magnetic heat of transformation occurs with decreasing nickel content so that the alloy with 0.35% Ni reveals only a single maximum composed of both curie peak and the maximum caused by the anomalous excess heat .

Similar effects have seen by W. Bendick (1978)[7] in other INVAR alloys such as ($Fe_{0.50}$ (Ni_x Mn_{1-x}) $_{0.50}$ or $Fe_{0.80-x}$ $Ni_x Cr_{20}$ [8] system .

The lattice parameter for $Fe_{0.65}$ $Ni_{0.35}$ alloy calculated from the (211) and (111) lines position using Bragg's Law for a cubic lattice with smallest error(∓0.1) due to incorrect values of 2 ϕ angle diffraction . Fig (3) present the change of the lattice parameter (a) versus milling time for the ball milling process of $Fe_{0.65}$ $Ni_{0.35}$ .The value of the lattice parameter increased from a= 3. 60 A° for 24 hr to a =3.79 A° for 96 hr . Such change of (a) when milling time increase was because of the small difference between the atomic sizes of the Ni and Fe atoms . The structural results fit well and agree with the data reported in Refs [11.12] .

To obtain a deeper understanding of the INVAR phenomena, it is helpful to see the influence of the milling time on the magnetic and thermal properties and collect microscopic information about the physical properties .More detailed discussion of these phenomena will be given in future work .

Table (1): Physical properties of the test Fe-Ni alloys at 296 K°

Proprettes	$Fe_{0.65}$ $Ni_{0.35}$	$Fe_{0.50}$ $Ni_{0.50}$	$Fe_{0.30}$ $Ni_{0.70}$
Average atomic mass (amu)	56.90	57.28	56.31
Lattice constant (A°)	3.596	3.586	3.586
Melting temp (K°)	1740	1710	1750
Curie temp. (K°)	528	810	840
Structure	FCC (gamma)	FCC (gamma)	BCC (alpha)
Saturation magnetic Moment (μ_B/atom)[9]	1.85	1.68	1.25

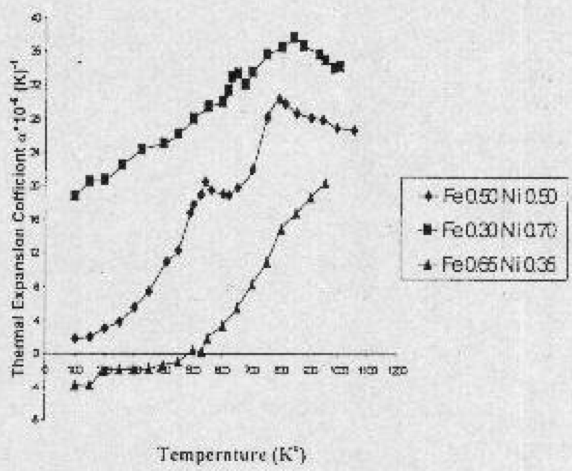

Figure (1): The Relation between thermal expansion coefficient (α) and temperature (K°)

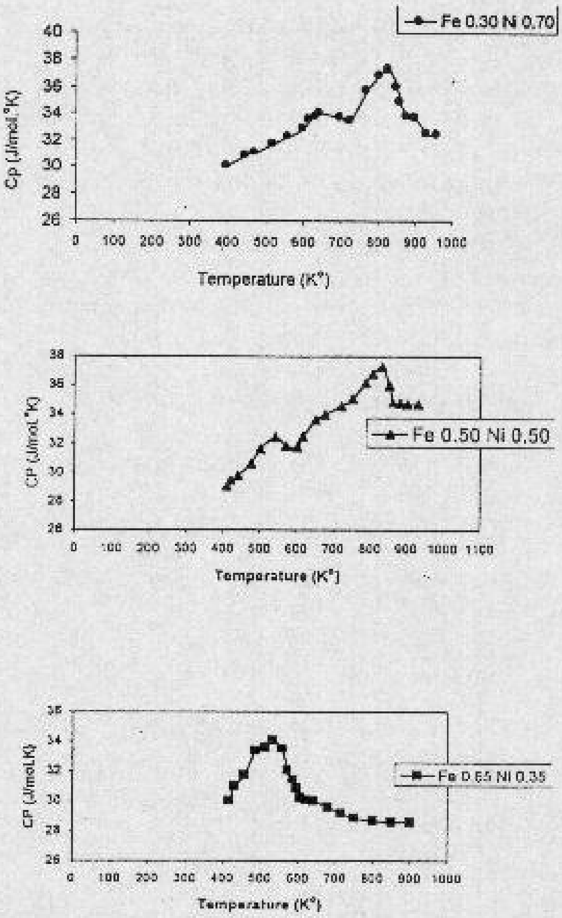

Figure (2): The retention between specific heat (C_p) and temperature (K°)

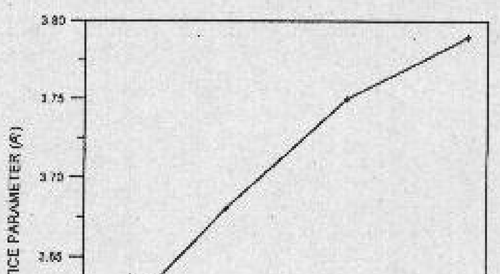

Figure (3): Lattice parameter of $Fe_{0.65}Ni_{0.35}$ alloy versus milling time

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الخلاصة

لغرض دراسة ظاهرة الانفار في سبائك العناصر الانتقالية تم اختيار سلسلة النظام الثنائي Fe-Ni لثلاثة نسب مختلفة، اجريت الفحوصات الحرارية والتركيبية للنماذج المحضرة ومن خلالها تم تحديد خاصية الانفار ، الاطوار الانتقالية ثم درجات حرارة كوري .

اكدت النتائج التجريبية بوجود شذوذ في معامل التمدد الحراري لسبيكة $Fe_{0.65}Ni_{0.35}$ حيث تم الحصول على قيم سالبة تحت درجة حرارة كوري (Tc) ، في حين اختفاء هذه الظاهرة عند النماذج الاخرى . اما النتائج التركيبية فقد اظهرت بوجود بنية تركيبية مكعبة متمركزة الاوجه (FCC) حينما تكون نسبة النيكل (35-50%) سرعان ما تتغير الى بنية تركيبية مكعبة متمركزة الجسم (BCC) حينما تكون نسبة النيكل (70%) .