

## Development of Smart Type of Ni-Cr Alloy as Self Thermally Controlling for Hyperthermia Cancer's Therapy

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### Abstract

In this study, developing method was used to synthesis a material benefit for cancer's therapy when heated remotely under alternated magnetic potential by hyperthermia treatments. Mechanical alloying processing (MA) with several milling times (12-72 h) used for producing four samples (1cm diameter x 0.3 mm thickness) of Ni-based alloy with various weights percentage of chromium (3-15 wt% Cr) in order to select which the smart type of them has the Curie temperature in a range of 41-46°C. then the samples were sealed in a vial under inert gas The samples were heat treated at 1200°C/3h in a furnace, then the samples were subjected to relaxation heat treatment at 600°C/2h and left for furnace cooled, followed by metallographic techniques. Characterization studies were evaluated for all treated samples including; Phase's analyses, microstructures images and the magnetic properties test.

**Keywords:** Hyperthermia, Thermosteds Materials, Curie Temperature and Mechanical Alloying.

تصنيع سبيكة ذكية من النيكل-كروم ذات خاصية السيطرة الذاتية لحدود معينة لدرجات الحرارة ملائمة للاستخدام

### الطبي في العلاجات السرطانية

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### الخلاصة

في هذه الدراسة، حضرت سبيكة ذكية من النيكل-كروم يمكنها توليد حرارة بحدود (41-46 م°، درجة حرارة كيوري) عند تعريضها لمجال مغناطيسي وهي مفيدة للعلاجات السرطانية عند استخدام طريقة العلاج بالحرارة العالية لقتل الخلايا السرطانية. تم استخدام طريقة التسبيك الميكانيكية لتحضير اربعة نماذج من سبيكة النيكل-كروم (بقياس 1سم قطر ويسمك 3ملم) وينسب وزنية مختلفة للكروم (3-15% وزنا) ولفترات زمنية مختلفة للطحن (12-72 ساعة). النماذج المحضرة توضع داخل انبوب زجاجي مفرغ من الهواء ثم تعامل حراريا بدرجة حرارة 1200 م°، تترك بعدها لتبرد داخل الفرن وبعد ذلك تجري عمليات الصقل والتنعيم. الفحوصات الطورية وصور التراكيب المجهرية والقياسات المغناطيسية أجريت على جميع العينات المحضرة من اجل معرفة أي من النماذج هو الافضل لتوليد درجة الحرارة المطلوبة للعلاج من الاورام السرطانية.

**الكلمات المفتاحية:** العلاج الحراري للسرطان، المواد المولدة للحرارة، درجة حرارة كيوري والتسبيك الميكانيكي

## Introduction

The interest in the bio-magnetism materials field, that the magnetic particles has a phenomena as (superparamagnetism and high saturation magnetization) leading to many medical applications such as drug delivery, imaging and hyperthermia cancer treatment. So it can define the hyperthermia as a method of thermal therapy for cancer's cells by exposure the body's tissue to relatively high temperatures (41-46°C) whereas this temperature range was find benefits for damage and/or kill tumor cells as well as an adjunct to cancer chemotherapy or radiotherapy based on the fact that cancer cells are more sensitive to temperature in these temperatures range when exposed to alternating magnetic field leading to generate heat by means of eddy currents and/or by the hysteresis losses achieved from magnetic thermoseeds or magnetic nanoparticles as heat sources (Masafumi *et al.* 2007, Hiroyasu *et al.*, 2010, Alexandru *et al.* 2011 and Robinson *et al.* 2011).

Ferrimagnetic materials considered as suitable thermoseed materials producing hysteresis heating by exposure them to an alternating magnetic field. The general idea regarding the relation between magnetic thermoseeds materials via Curie temperature can

Briefly illustrated as that, the thermoseeds material means the material which has the behavior for converting from ferromagnetic to paramagnetic at limited range, and after that it loose the magnetic behavior when the magnetic potentials increased. Therefore, it is important to obtain an alloy with low Curie temperature whereas a magnet will lose its magnetism property if heated above the Curie temperature (Akin *et al.*, 2009, Radev *et al.* 2012 and Karen *et al.* 2012).

Nickel-chromium ( $Ni_{1-x}Cr_x$ ) particles with varying compositions have been investigated as thermoseeds for use in localized self controlled hyperthermia

treatment of cancer in order to prevent over heating of normal cells. The mechanical alloying processing (MA) was find suitable for synthesis the Ni-Cr alloy,

its mechanism involved (the grinding to a fine powder by using ball miller with aid of liquid agent, isostatic pressing and sintering processing (Chicinas *et al.* 2003, Giovanni *et al.* 2009 and Robinson *et al.* 2011)

In this research, preparations of Ni-Cr alloy with different composition of chromium (5-20wt. %) using mechanical alloying processing method to obtain the suitable Curie temperature when the alloy samples explored under magnetic field.

## Materials and Methods

The starting materials which required to synthesis Ni-Cr alloy at different composition were introduced as high purity (99.98 Fluka, Germany) for both nickel and chromium metals. Four alloy samples with various wt% of chromium were prepared in order to evaluate the suitable specimen which has the Curie temperature in a range of 42-48°C.

In this study, nickel-chromium alloy was prepared by mechanically alloying process, which involves the addition of 5-20 wt% chromium to the nickel based powder. The particle sizes of the starting powders were 74 and 62  $\mu m$  respectively. The powders were mixed and subjected to mechanical alloying process in a miller with hardened steel balls at 40:1 weight ratio of ball/powder, Several milling times were used from 10-72 h. the resulting powders were mixed with 1.0 wt.% polyvinyl alcohol (PVA) as a binder and uniaxially pressed into pellets at a pressure of 3.0 ton/cm<sup>2</sup> to make samples with 1cm diameter and 0.3 mm thickness, and then the samples were sealed in a vial under inert gas The samples were heat treated at 1200°C/3h in a furnace, then the samples were subjected to relaxation

heat treatment at 600°C/2h and left to furnace cooled.

Metallographic techniques on samples including grinding and polishing to mirror face by using different grades of Silicon Carbide (SiC) papers with varying polishing pastes. The polished samples were subjected to etching process before taking the microstructure images by using Mable solution (CuSO<sub>4</sub> + HCl + H<sub>2</sub>O). Characterization studies were evaluated for all treated samples including; Phase's analyses which were measured by using the X-Ray **diffraction** technique at scanning speed (0.1° /min). Microstructures images were evaluated by optical microscopy and/or SEM technique. The magnetic properties such as receptivity, **coerecivity**, and

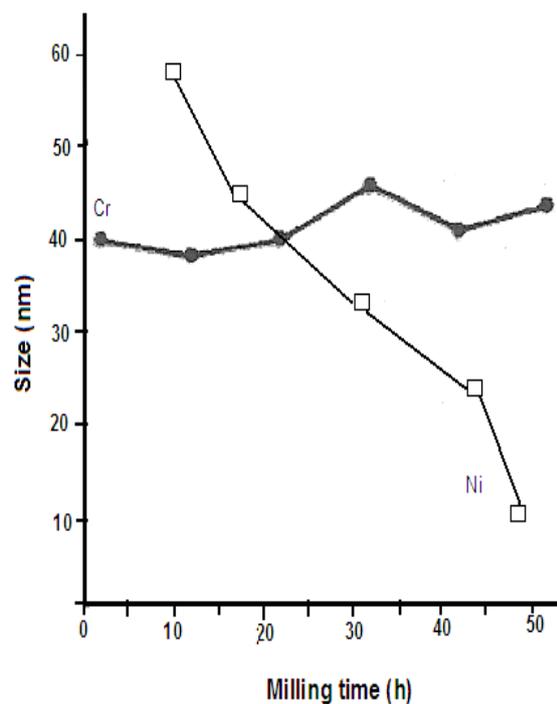
The effects of milling times on the particle size of the nickel and chromium powders were showed in fig.1, which revealed the decreases in particle size of nickel from 58nm/12 hours of ball milled to 10nm/48hours. While relatively lower varying in chromium particle size was observed at the same increasing of ball milling times. This probability belonged to the formation of Ni/Cr solid solution during the ball milling processing due to the chromium particles could be diffused into the nickel lattice and forms a solid solution.

Fig.2 showed the XRD patterns of Ni and Cr powders at various periods of mechanical milled (MA) compared with powders unmilled. This figure observed that Ni peak at (220) was shifted to lower angle when increased in milling time. The peaks (200) for both Ni and Cr decreased and the width increased via the milling time. Characteristic peaks of Face center cubic (fcc, Ni) and body center cubic (bcc, Cr) were observed after 12 hours of ball milling showing the refinement evaluations. For that, when the milling time increased, the diffraction peaks behaviors were broadened via intensity decreased. Ni

saturation magnetization will be measure by using vibrating sample magnetometer technique (VSM).

### Results and discussions

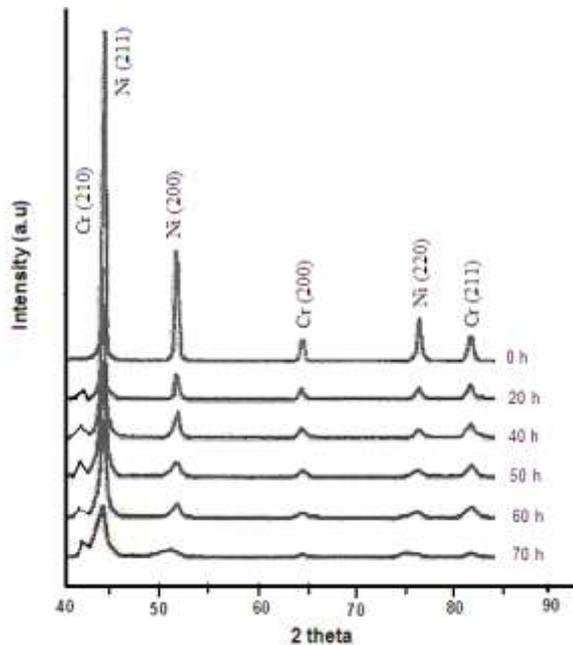
Various types of Ni-Cr alloy samples were prepared with varying chromium's weight percentage relating to nickel (as the matrix alloy) in order to find the specific composition to give the suitable Curie temperature in the range of 41-46°C. Mechanical milling alloying technique was used for preparing samples followed by recrystallization temperature at 650°C for 3 hours in a sealed quartz tubes (for relaxes the internal stresses and favors' the solid-state reaction). The reaction becomes significant after 48 hours of milling and subsequent heat treatment.



(Cr) solid solution was occurred due to the chromium diffused in (fcc) nickel matrix. Furthermore, the (fcc) chromium was reduced in its intensity and remains in visible along

**Fig. (1) Mean Particle Size at Different Milling Time for Ni-Cr Powders**

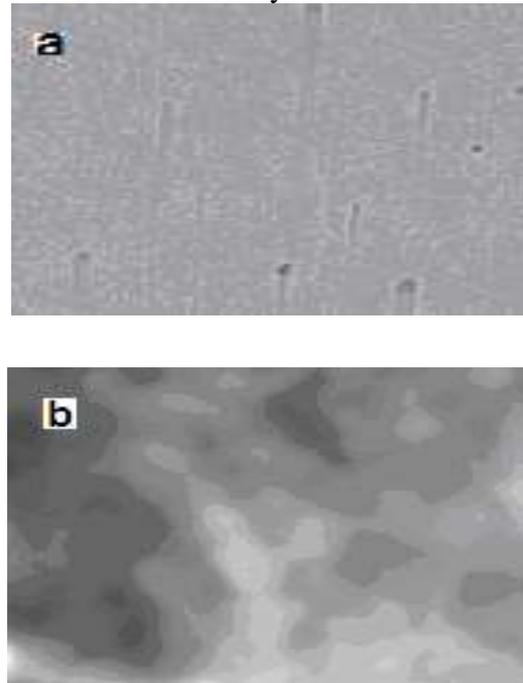
ball milling for 30 hours. This result leading to confirm that the solid state alloy formation occurs during milling, also it was observed that the chromium at (211) peak completely disappeared after 36 hours of milling due to formation of the final Ni(Cr) phase. Preparation's mechanism of the alloy was found to be depending on its morphological and/or its initial powders fabrication. The mechanical alloying processing of metals was to be a lamellar process, which that the milling process was found affected by decreased the lamellar



**Fig. (2) XRD Pattern for Ni-Cr Powders at Various Periods of Millings**

spacing, for that the increased contact between surfaces of powder particles could occurred when the initial powders have a small particle size and/or narrow size distributions leading to decrease the processing time. It was observed that the crystalline size decreased to less than 50nm after first stage of milling, further the crystalline refinement took place slowly after 40 hours of milling. The optical microscopy was used to analyze the alloy samples after mechanical milling for 36 hour (figure 3 a) and after 48 hour (fig.3 b) which revealed that morphological and structural features as

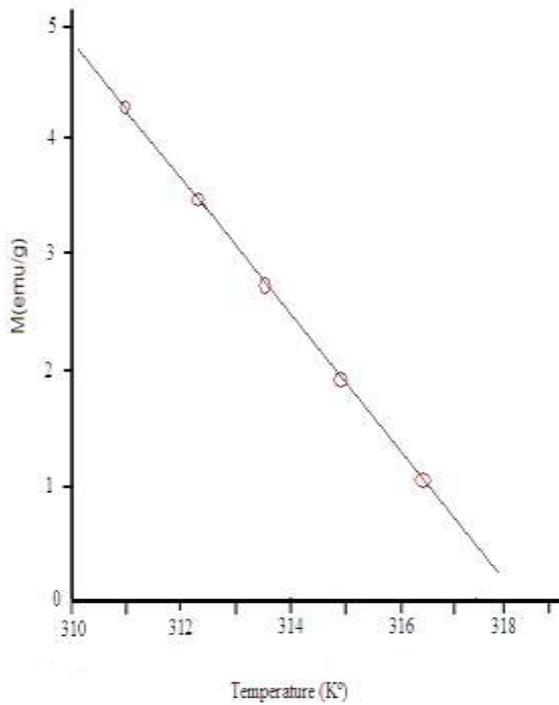
homogenous and fully diffused chromium in the Ni matrix forming the solid state alloy.



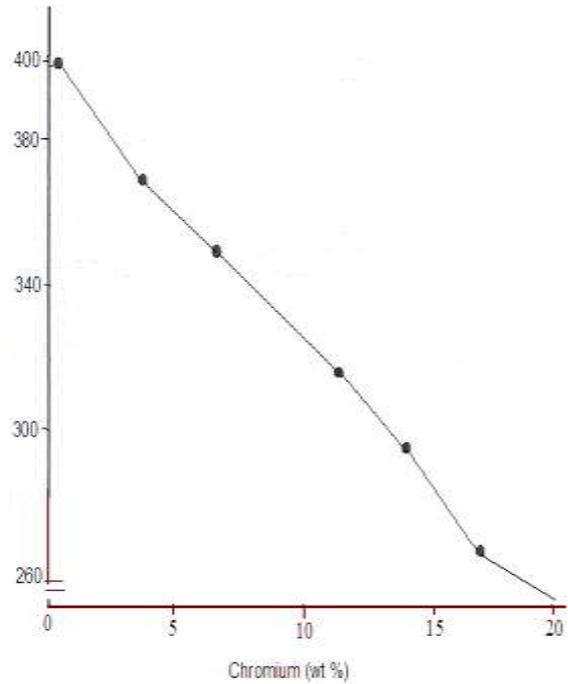
**Fig. (3) Micrograph Images of Ni-Cr Alloy after Mechanical Milling**

**(a) 36 h and (b) after 48 h**

The Magnetization measurements were carry out to obtain the specimen's Curie temperature ( $T_c$ ), which determined as showed in fig. 4 that the sample's behavior found to decrease in spontaneous magnetizations via increased in temperatures. The specimen (Ni-10Cr), paramagnetic state appeared via spontaneous magnetization at the temperature of 317 K°.



**Fig. (4) Effect of Spontaneous Magnetization on Curie Temperature for Sample Ni-10Cr**

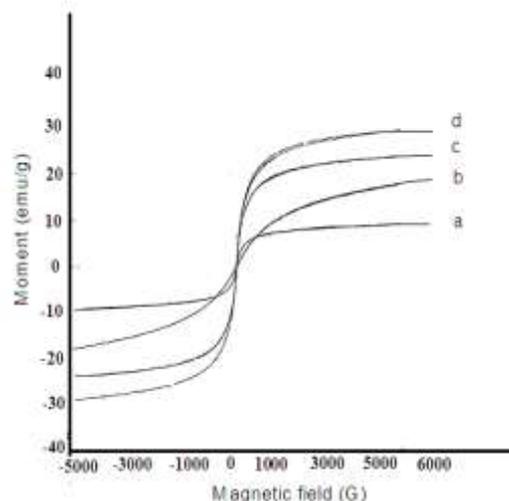


**Fig. (5) Curie Temperature Versus wt% Chromium Concentration**

Fig.5 showed the effect of chromium concentration on Curie temperatures, so the decreased in Curie temperature proportionally increased with chromium concentration in the alloy

rate increased verses saturation decreased when the chromium wt% increased in the Ni-based alloy due to formation of a surface spin structure led to predict the significant selected of processing method for getting preferred magnetic properties, this fact also confirmed by Wilhem *et al*, 2007

Fig. 6 showed the hysteretic loops of different chromium composition in nickel based alloys as a function between mass magnetizations measured at the room temperature and the applied magnetic field, which revealed initial rapid increased in magnetization via slower increased in the H value, led to predict that the alloy response behavior as typical



**Fig. (6) Hysteric Curves for Different Types of Ni-Based Alloys Specimens (a) Ni-5Cr, (b) Ni-5Cr-4Ti, (c) Ni-10Cr and (d) Ni-15Cr**

## Conclusions

From this study, can be concluded that the mechanical alloying technique (MA) considered as a simple technique for producing various types of alloy systems. It results in loss of the metastable effects, so it was use in the bulk shapes and useful to produce materials at large quantities. By controlling the milling conditions (weight ratio of ball / powder and milling energy), the solid solution of metal powders could be synthesized.

From the comparison of the materials at long times of milling via normal intensities, it was observed that the composition ranges of the bcc and of the bcc and fcc phases were highly extended verses their equilibrium ranges. So when using the high milling intensity, the phase (bcc) was lost its stabilized via the phase (FCC) leaded and shifted to low concentrations of nickel

The alloy's Curie temperatures found in linear decreases via increases in chromium concentration, so the Ni-Cr alloys considered as perfect for self magnetic controls making their benefit to be use in the hyperthermia treatments of tumors and to avoid tissue's overheating.

## References

**Alexandru., J.**, Mirela, M; Rami, S; and Eros, A. (2011) "Low Curie Temperature In Fe-Cr-Ni-Mn Alloys" U.P.B. Sci. Bull., Series B, 73 (1), 42-49.

**Akin, Y** Obaidat, M; Issa, B. and Haik Y. (2009) "Ni<sub>1-x</sub>Cr<sub>x</sub> Alloy for Self Controlled Magnetic Hyperthermia" Crys. Res. Tech. 44 (3), 386-390.

**Chicinas, I;** Pop, V; Isnard, O; Breton, J. and Juraszek, J. (2003) "Synthesis and Magnetic Properties of Ni, Fe Intermetallic Compound Obtained by Mechanical Alloying" J. Alloys and Comp. 352 (2), 34-40.

**Giovanni, B;** Giada, L. and Costanza, R. (2009) "Hyperthermic Effect of Magnetic Nanoparticles under Electromagnetic Field" Processing and Application of Ceramics 3 (1), 103-109.

**Hiroyasu, K;** Hideki, H; Yoshinaka, S. and Tada, K. (2010) "In Vitro Biocompatibility of Ni-Free Ti-Based Shape Memory Alloys for Biomedical Applications" J. Mat. Trans, 51 (10), 1944-1950.

**Karen, S.** (2012) "Thermosensitive Magnetic Nanoparticles for Self-Controlled Hyperthermia Cancer Treatment" J. Nanomed. Nanotech. 3 (6), 73-78.

**Masafumi, M;** Tokiko, H; Kiyoshi, Y; Yuji, S; Takayuki, H. and Yuji, K. (2007)"Evaluation of Biocompatibility for Titanium-nickel Shape Memory Alloy in Vivo and in Vitro Environments" J. Mate. Trans, 48 (3), 352 – 360.

**Radev, D.** (2012) "Nickel-Containing Alloys for Medical Application Obtained by Methods of Mechanochemistry and Powder Metallurgy" Int. SchoL. Res. Article ID 464089

**Robnson (2011).**impreg nation of bio-oil Vol .6(u) pp. 4747-4761 bioresources.

**Wilhelm, W.W.** Corn Stover to sustain soil Organic Carbon further Biomass Supply Agronomy Journal Vol.9 (2007).