Fabrication of TiC Particulate Reinforced Ni-50Fe Super Alloy Matrix Composite Powder by Mechanical Alloying

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ABSTRACT

Mechanical alloying is a powder metallurgy processing technique involving cold welding, fracturing, and rewelding of powder particles in a high-energy ball mill. In this work, NiFe-TiC composite powders were prepared by mechanical alloying process using planetary high-energy ball mill. The effect of TiC addition by weight percent on the NiFe solid solution formation, grain size, lattice parameter, internal strain and hardness of composite powders was investigated as a function of milling time, t, (in the 0-25 h range). Microstructural and phase characterizations investigationof the mechanically alloyed powders were carried out using X-ray diffractomejhghfter (XRD) and scanning electron microscope (SEM). The results showed that the brittle particles of TiC accelerate the milling process by increasing the matrix deformation and enhancing the welding and the fracture of particles. We also found that the NiFe solid solution formation occurred atearlier time of mechanical alloying with increasing the TiC content. Moreover, it was shown that with increasing theTiC by weight percent, smallercrystallite size and more hardness are obtained after mechanical alloying.

1- Introduction

The ever-increasing need for applications of materials especially in aerospace and automobile industries has resulted in development of composite materials. Among these materials metal-based composites formed by a hard ceramic reinforce in an alloy or soft metal matrix can be mentioned. Metal-based composites are a combination of metal properties (flexibility and density) with ceramic quality (strength and high module) which results in higher strength under cutting and pressure as well as higher applicability [1].

Super alloys have a wide range of various applications. The most important of these applications are corrosion and heat resistance which are specifically useful in gas turbines of aircrafts, vapor turbine plants, typical automobile parts such as fume valves, casting molds,nuclear plants systems and chemical and petrochemical industries [2].

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Fi-Ni based superalloys can be produced by electrodeposition [2, 3, 4], [5], hydrogen plasma reaction [6], gas condensation [7], thermal evaporation [8], rapid solidification [9] and mechanical alloying [10-16]. Mechanical alloying which is a dry and high energy milling process has been increasingly attracting attention in recent years due to the wide range of materials which can be synthesized: amorphous, quasi-crystalline and nanocrystalline phases, extended solid solutions, alloys of immiscible elements and different kinds of compounds and composites. This process involves repeated fracture and welding of particles which results in deformation and reduction of particles size. Finally alloying takes place by interdiffusion across welded interfaces of grains.Moreover, mechanical alloying is positively used in production of nanocrystalline materials with nanostructure. Mechanically alloyed powders have properties such as small grain particles and flexibility in selection of alloying [17].

2- Research methodology

The starting materials used in the present research include nickel powder (mean particle size 100µm, 99.5% purity, LSM Co.), iron powder (mean particle size 150µm, 99.2% purity, Hoganass Co.) and TiO2 powder (mean particle size 200µm, 99.8% purity, Alfa Aesar Co.). The form and size of particles are shown in fig 1. The mixture of FeNi-XTiC(X=0, 5, 10, 20, 30 Wt% TiC) was milled for 1, 3, 5, 7, 10, 15 and 25 hours by a high energy planetary ball mill rotating at the speed of 600 rounds per minute (600rpm) in high-chromium hard steel vials with steel ball bearings with 20mm diameter. To prevent oxidation phenomenon, the vials were positioned under argon atmosphere. The ball-to-powder ratio (BPR) was 20:1.XRD experiments were performed by a Philips PW1800 diffractometer using CuKα radiation. Morphology and microstructure of milled powders were studied by scanning electron microscopy (SEM, LEO 435VP and VEGA TESCAN). Crystallite (grain) size and lattice strain were measured based on Williamson-Hall method. Vickers microhardness test was performed on 10-hours-milled powders by ERNSTLeitz GMBH WETZLAR tester under 50gr load.

Titanium carbide (TiC) with great hardness and thermal stability is an appropriate reinforce in iron-based composites. These composites are generally produced by powder metallurgy method which involves addition of TiC powder to iron powder. Also, due to its high melting point (3060 °C) and low density (4.9 gr/cm3), it is regarded one of the suitable materials in high temperature applications [18].

Some researchers have used different methods for producing FeNi-based superalloys reinforced by ceramic particles and studied their properties [19-21]. However, studies about FeNi-based superalloys reinforced by TiC particles are not sufficient. On the other hand, one of the advantages of mechanical alloying in production of metal-based composites is the uniform distribution of reinforce in metal matrix.FeNi-based superalloys reinforced by titanium carbide are currently used in high temperature applications where corrosion and wear are the major sources of material failure.

3- Results and discussion

X-ray diffraction pattern for powder composition of Fe-50Wt%Ni after different milling times is shown in fig 2. As can be seen, with increase of milling time all the peaks are broadened and their intensity is reduced which shows steady decrease of grain size and presence of lattice strain.The diffraction lines shown in zero hours is assigned to iron with body center cubic (BCC) and nickel with face center cubic (FCC). With increase of milling time up to 7 hours, both iron and nickel peaks show themselves but no shift in their position is observed; only their intensity is reduced and their width increases. After 10 hours of milling the peaks corresponding to (110) and (200) planes which are related to the BCC α-Fe disappear and only the reflections corresponding to FCC phase are present with the peaks slightly shifted towards lower angles.This implies the formation of solid solution of iron in nickel. In other words, omission of the iron peaks and shift

of nickel peaks reveals that iron atoms have been diffused into nickel matrix and since the atomic radius of iron (0.126nm) is larger than that of nickel (0.124nm), the lattice is expanded and FCC solid solution of γ(fe, Ni) is produced. The resulted solid solution is called taenite. With acceleration of milling time to 25 hours, the diffraction lines are widened but no conspicuous shift of peaks is observed which shows that the grains have been significantly refined. It should be added that residual stresses in materials cause distribution of tension and compression stresses which results in the widening of the peaks.Microstresses in crystals stem from sources such as vacancies, defects, shear plates, thermal expansion and contractions. Those powders which have not undergone annealing have higher residual stress and more widening in peak lines [22].

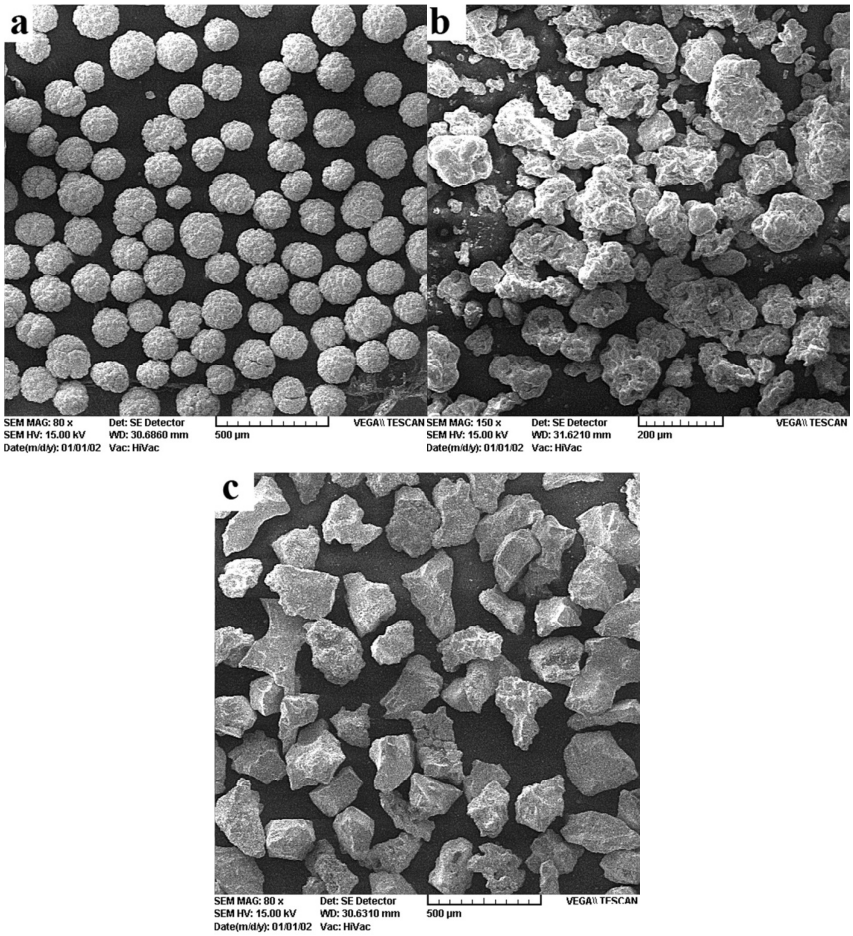

Fig.1. Size and morphology of starting materials (a) Ni, (b) Fe, (c) TiC

Formation of FCC solid solutionγ (fe, Ni) called taenite for $Fe_{50}Ni_{50}$ powder composition resulted from mechanical alloying after 24 hours of milling [23], after 50 hours [24] and after 2 hours [25] have been reported by different researchers. X-ray diffraction pattern of $Fe_{50}Ni_{50}$ powder composition after addition of TiC at different milling times is shown in fig 3. Fig 3a shows X-ray diffraction pattern of addition of 5Wt% of TiC and fig 3b shows diffraction pattern of addition of 10 Wt% of TiC. As can be seen, in both figures with increase of milling time the intensity of the peaks is reduced and the peaks

are broadened which is due to reduction of grain size and formation of lattice strain. In fig 3b, decrease of peaks intensity and widening of the peaks is more than in fig 3a. For example, this trend is quite conspicuous after 5 hours of milling for both figures. Also, by comparison with fig 2, it can be concluded that formation of solid solution for Ni-Fe-10 Wt% TiC occurs at shorter milling times.

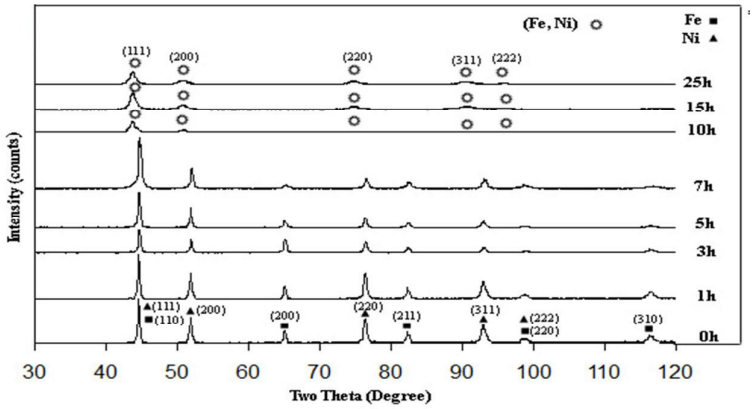

Fig.2.XRD patterns of Fe-50wt%Ni after different milling times

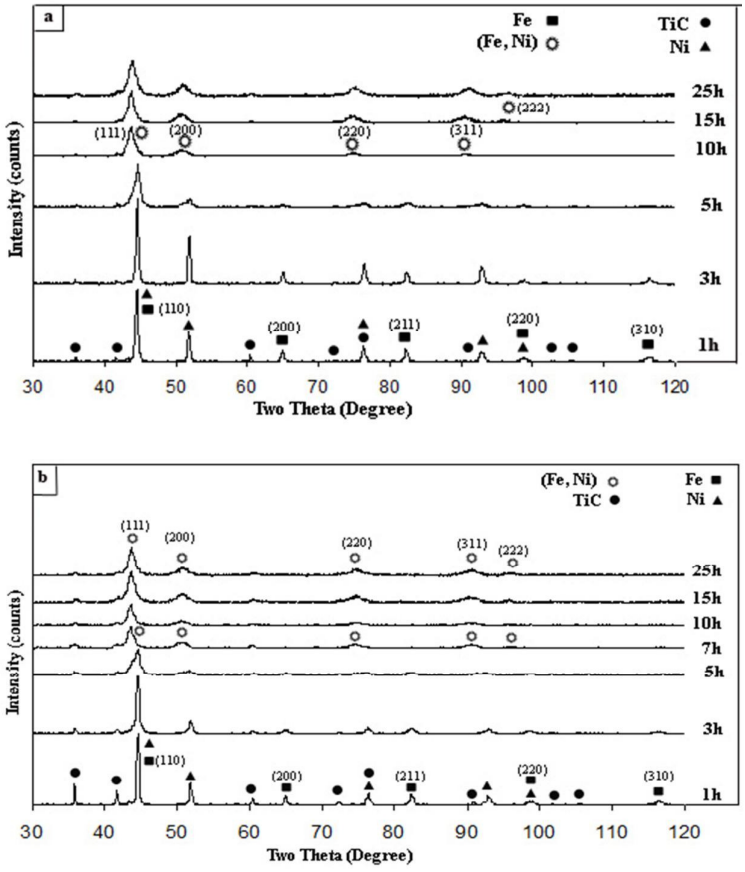

Fig.3. XRD patterns of $Ni_{50}Fe_{50}$ together with,a) 5Wt%TiC, (b) 10Wt%TiC

This means that in fig 3b, after 7 hours of milling the iron peaks have been omitted and the peaks corresponding to FCC phase have shifted towards smaller angles, whereas in fig 2 after 7 hours of milling, the iron peaks are still present and there is no shift of peaks.

Mechanical alloying stages in systems including ductile-brittle parts involves plastic deformation of the ductile part, breaking of the brittle particles, cold welding and fracture of deformed particles [26]. The presence of brittle phase particles increases local deformation around the ductile phase. Moreover, hard particles increase work hardening rate of metal particles. Therefore, fracture process in ductile-brittle systems occurs sooner than in ductile-ductile ones. On the other hand, initiation of alloying is due to a combination of parameters such as decrease of diffusion distance, 26]. (interlamellar distance), increase of lattice defects density and any heating that may have been generated during the milling operation. In other words, increase of hard particles (reinforcement) weight percent results in more interaction between dislocations and hard particles which accelerates the speed of alloying and particle size reduction processes [17]

Fig 4 shows lattice parameter variations versus milling time for powder composition NiFe-0, 5, 10Wt% TiC, which was calculated by Nelson and Riley equation as follows [22]:

$$a = a_0 + a_0 k\left(\frac{cos^2\theta}{sin\theta} + \frac{cos^2\theta}{\theta}\right) \qquad (1)$$

Where a is the lattice parameter calculated for every line in cubic materials, a_0 is the actual value of lattice parameter, θ is diffraction angle and k is the constant. By drawing the value of a versus $\left(\frac{cos^2\theta}{sin\theta} + \frac{cos^2\theta}{\theta}\right)$, a line is obtained whose slope and y-intercept show the value of a_0k and a_0, respectively. It should be added that the above mentioned equation is more applied in cubic materials. As can be seen, with increase of milling time, lattice parameter increases from 0.3528nm for pure nickel after zero hours of milling to around 0.3607nm for NiFe-0, 5, 10Wt% TiC after 25 hours of milling. The sudden increase of the lattice parameter in the range between 5 to 10 hours of milling is observed in all cases and after that the lattice parameter increases with a lower slope.In the range 5-10 hours of milling while pure iron disappears, $Ni_{50}Fe_{50}$ alloy is progressively being formed, but in the range 10-25 hours when only one alloy phase is present, the increase of lattice parameter is not the same as before. Also, regarding NiFe-0Wt% TiC sample, lattice parameter is constant up to 5 hours of milling with no conspicuous changes. With increase of TiC up to 5Wt% and milling for 3 hours, lattice parameter increases a little. With increase of TiC to 10Wt% after 5 hours of milling, the increase of lattice parameter is more than the two previous cases. The increase of lattice parameter with increase of milling time is due to the irregularity of alloy. Similar results about increase of lattice parameter with milling time for $Ni_{50}Fe_{50}$ [23] and FeAl [27]. Also, as was mentioned before, addition of a hard phase such as TiC to the system causes the increase of crystalline defects and consequent deformations caused by such defects. Therefore, in the areas surrounding crystalline defects, the distance between crystalline phases increases and causes the shift of peaks toward smaller angles and ultimately the increase of lattice parameter. For comparison, the lattice parameter obtained for $Ni_{50}Fe_{50}$which had been produced by various researchers through mechanical alloying method can be mentioned: this parameter is 0.359nm after 50 hours of milling [24], 0.3590nm after 1 hour [28] and 0.3595nm after 50 hours of milling by using planetary ball mill.

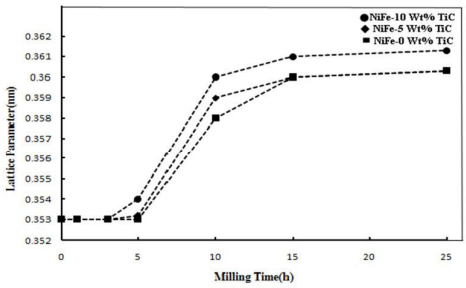

Fig.4. Lattice parameter versus milling time for NiFe-TiC

Crystallite size variations and lattice strain versus milling time for powder mixture NiFe-0, 10, 5Wt% TiC is shown in fig 5. It is obvious that crystallite size decreases in all cases (both the samples with TiC and those without TiC) with increase of milling time (fig 5a). The intensity of decrease in crystallite size is high at first and then declines. This decrease of grain size is accompanied by increase of internal strain (fig 5b). As can be seen, with increase of TiC up to 10Wt%, the decrease of crystalline size is more intense. Crystalline size starts from 40nm at milling for zero hours and after 25 hours of milling reaches to 12nm, 10nm and 8nm for NiFe, NiFe-5Wt% TiC and NiFe-10Wt% TiC, respectively. Internal strain starts from 0.075% at milling for 0 hours, and after 25 hours of milling increases to 0.4%, 0.7% and 0.9% for NiFe, NiFe-5Wt% TiC and NiFe-10Wt% TiC, respectively. The reason for increase of lattice strain with increase of TiC could be the fact that with increase of TiC plastic deformation and work hardening will increase and consequently a higher density of defects especially dislocations is developed. Various reports about crystalline size and lattice strain for NiFe have been presented, for instance one can refer to 12nm crystalline size and 0.41% internal strain after 50 hours of milling in a planetary ball mill [23], 10nm crystalline size and lattice strain 0.5% after 50 hours of milling in a planetary ball mill [24], 8nm crystalline size after 48 hours of milling in a planetary ball mill [25], 14nm crystalline size and 0.5% lattice strain after 400 hours of milling in a horizontal low energy ball mill [14], 15nm crystalline size for NiFe alloy prepared by chemical excitation method [28] and 45nm crystalline size obtained by gas condensation method [7]. Morphology and grain size were studied by scanning electron microscopy (SEM). Fig 6 shows morphology and particle size of $Ni_{50}Fe_{50}$ after different milling times. As can be seen, the shape and size of particles are different in various milling times. Fig 6a shows the shape and size of particles after milling for 1 hour; the particles have a flake shape. In the initial stages of mechanical alloying due to the trapping of the powder particles between the balls and the collision of the particles with each other, the particles get flattened to take flake/platelet shapes. In other words, in primary stages of mechanical alloying, because of microforging of ductile components, the particles are deformed and take a flake-to-pancake shape [17]. The above mentioned trend is more obvious after 3 hours of milling (fig 6b). As can be seen, deformation and flaky shape of particles is quite observable. In the next stage these flat/flaky particles are welded together and make a compound with layered structure, which is a normal state for the materials prepared by mechanical alloying of ductile-ductile or ductile-brittle systems. Increase of particles size is also observed in this stage. With increase of mechanical alloying duration, the hardness and consequently (brittleness) increase. Fig 6c shows alloying after 5 hours which involves cold welding and fracture. With increase of alloying duration and continuous repetition of cold welding and fracture the particles are deformed and as a result particles with equiaxed dimensions are formed. Fig 6d shows alloying after 7 hours where the flaky shape of particles changes into equiaxed dimensions. But some particles with flaky shape are still observed. Fig 6e shows alloying after 25 hours: most particles have equiaxed dimensions and a consistent distribution of particles is observed which is due to the balance between cold welding and fracturing processes and reveals that the system has reached a steady state or equilibrium. Some researchers [23, 25] have reported similar results for NiFe alloy prepared by mechanical alloying method. Fig 7 shows morphology and particles size NiFe-10Wt% TiC versus different milling times. Fig 7a shows mechanical alloying after 3 hours in which the particles get flattened. With increase of milling duration to 5 hours (fig 7b) the particles get equiaxed dimensions. This reveals that addition of a brittle phase (TiC) adds to the speed of alloying process. Fig 7c shows alloying after 25 hours. As can be seen, the particles get smaller and their

shapes are similar. Also, a kind of agglomeration of particles is observed. The reason for the trend which was observed in fig 7 can be explained in this manner: the presence of reinforcement particles in powder mixture of Ni and Fe changes the mechanical alloying system to ductile-brittle state. For better understanding it is necessary to give a brief explanation about the way the process is carried out in this system at different stages of milling. In the primary stages of milling while the ductile particles are deformed, the brittle ones get fragmented. Following that, while the ductile particles start to weld, the brittle particles are trapped between ductile particles. Therefore, the fragmented reinforcement particles will be located in interfacial boundaries of welded metal particles and the product is a composition (compound) of particles. If welding is the dominant mechanism in the process, the platelet particles are accumulated on each other and get deformed. These phenomena (deformation, cold welding, particles distribution) results in hardness of materials and increase of fracturing process. Moreover, these phenomena help the formation of particles with equiaxed dimensions, too. Then by reaching a balance between cold welding and fracturing mechanisms, randomly oriented composite particles with interfacial grain boundaries will be formed. In the final stages (steady state), microstructure is favorably improved and interfacial boundaries are no longer visible under an optical microscope. Similar results about Al-AlN [29, 30] and Al-SiC systems [31] have been reported by other researchers. To explain the effect of reinforcement on mechanical alloying process it should be mentioned that cold welding process which occurs in mechanical alloying is due to cold deformation. There is a critical deformation above which cold welding phenomenon occurs and below which there is no welding of particles and only work hardening occurs. The presence of reinforcement particles between the particles which are welding causes the increase of local deformation around reinforcement particles. Increase of local deformation improves cold welding process. On the other hand, with more deformation applied by reinforcement particles work hardening and hence fracturing process will increase. Such acceleration in cold welding process which is due to the presence of reinforcement particles may be a reason why the whole process of mechanical alloying happens in a shorter time. Another possible reason could be that hard and small brittle particles in the matrix act as milling agents and increase system energy, therefore the milling time needed for reaching steady state will decrease [29]. Table 1 shows the effect of addition of 0, 5, 10, 20 and 30Wt% TiC to NiFe on lattice strain and crystalline size after 10 hours of mechanical alloying. Also, in order to study the effect of TiC on hardness of 10-hour-mechanically alloyed powders, these powders were warm mounted and Vickers microhardness test under 50gr load was performed; the results are presented in table 1. As can be seen, with increase of TiC, internal strain and hardness increase, whereas the particle size decreases. The mechanism of hardening of metals and alloys by deformation grain refinement and distribution of solid particles is widely known. Mechanical alloying causes a great deal of deformation, decrease of grain size to nanoscale and, depending of process parameters, a very minute distribution of oxides, carbides and nitrides in metal structures. Also, due to intensive deformation, a high density of dislocations occurs which can be a reason for increase of hardness with addition of TiC. In other words, increase of hardness of mechanically alloyed powders with increase of the amount of TiC could be due to two reasons: the effect of mechanical alloying process on the alloy matrix and the effect of reinforcement.

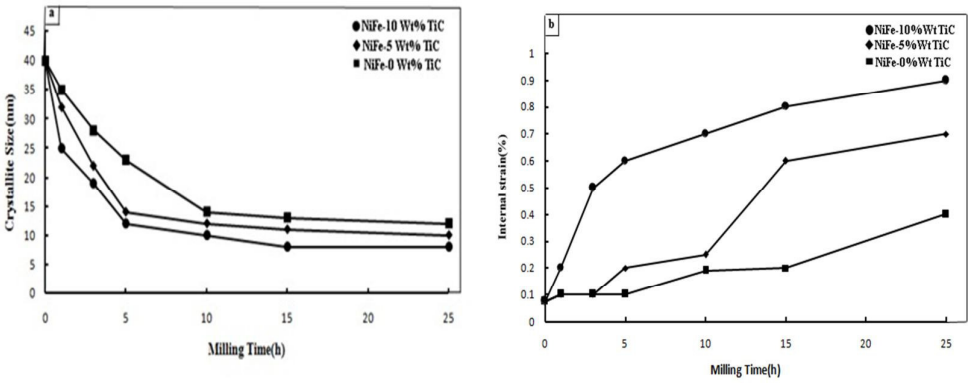

Fig.5. Variation in (a) crystallite size and (b) internal strains of milled powder with milling time for NiFe-TiC

4- Conclusion

Ni and Fe powders and TiC particles were alloyed by milling and then the effect of the amount of TiC weight percent on mechanical alloying, hardness and microstructure of mechanically alloyed powders was investigated and the following results were observed:

1- The mixtures of Ni-50Fe are completely dissolved after at least 10 hours of mechanical alloying in a high energy mill and FCC solid solution is produced.

2- The presence of reinforcement particles adds to the speed of mechanical alloying process due to increase of deformation in the matrix, so that by using 10% TiC in the composition, alloying time decreases down to 7 hours.

3-With increase of TiC amount, the matrix grain (crystal) size will decrease.

4-The presence of TiC particles in the matrix increases hardness and this trend accelerates with increase of TiC amount

Table1. The effect of TiC addition to NiFe on grain size, internal strain and hardness of mechanical alloyed powder after 10h

Sample	Grain size(nm)	Internal strain (%)	Micro Hardness($HV_{0.05}$)
NiFe-0Wt%TiC	$14^{+}_{-}2$	0.19	$280^{+}_{-}30$
NiFe-5Wt%TiC	$13^{+}_{-}1$	0.25	$720^{+}_{-}40$
NiFe-10Wt%TiC	$12^{+}_{-}1$	0.7	$850^{+}_{-}25$
NiFe-20Wt%TiC	$10^{+}_{-}1$	0.9	$930^{+}_{-}15$
NiFe-30Wt%TiC	$8^{+}_{-}1$	0.98	$1017^{+}_{-}20$

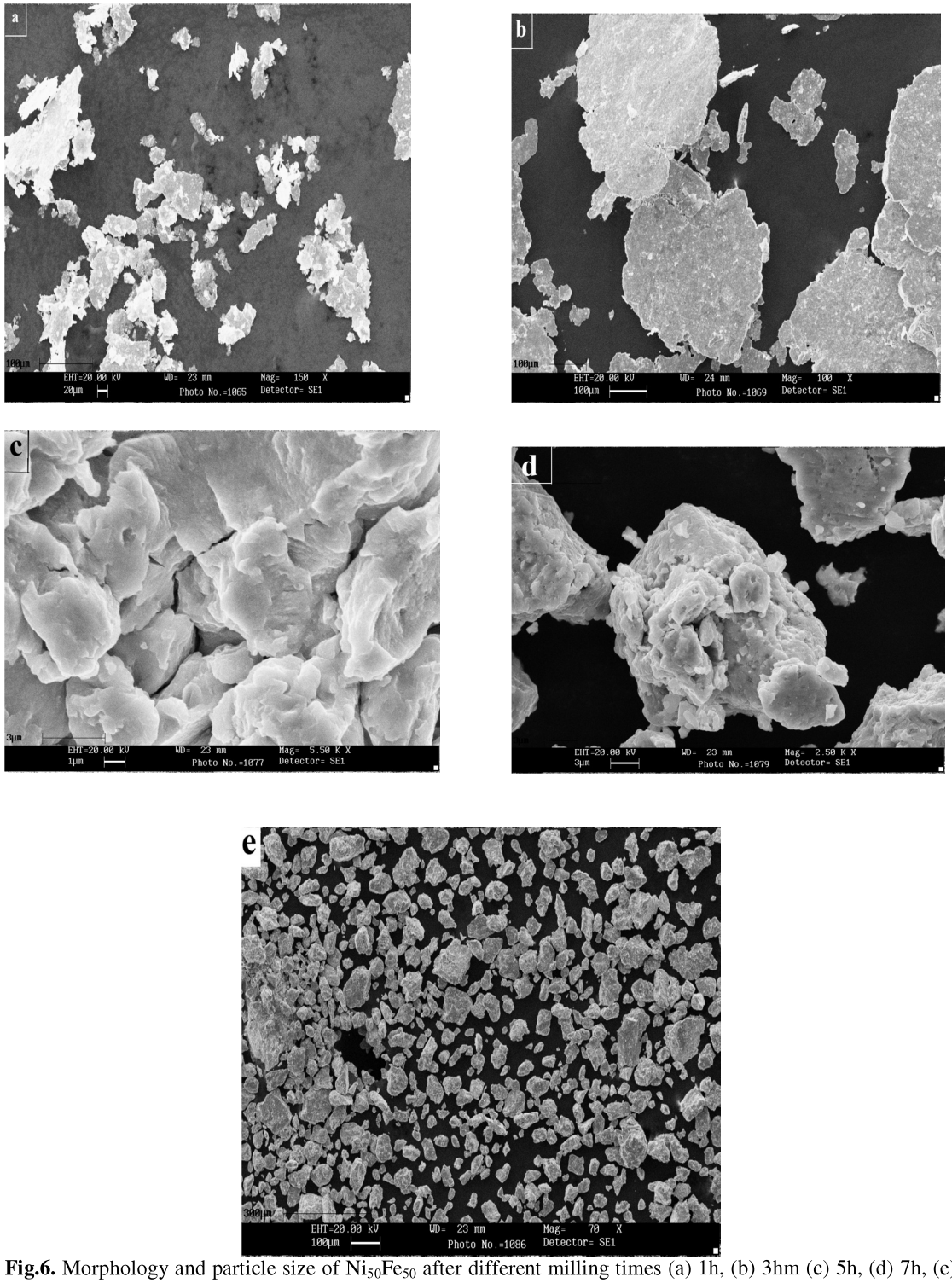

Fig.6. Morphology and particle size of $Ni_{50}Fe_{50}$ after different milling times (a) 1h, (b) 3hm (c) 5h, (d) 7h, (e) 25h

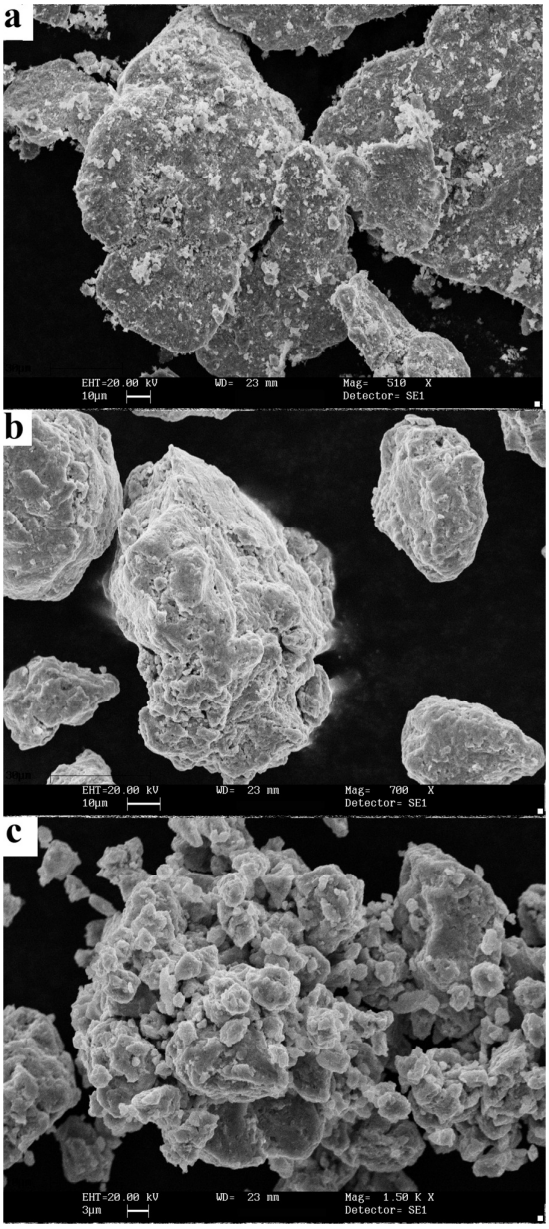

Fig.7. Morphology and particle size of NiFe-10Wt%TiC after different milling time, (a) 3h, (b) 5h, (c) 25h

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