SVOA Materials Science & Technology

ISSN: 2634-5331

ScienceVolks Open Access

Research Article

Synthesis of Intermetallic Compounds in the Ni-Cr-Si System by Mechanical Alloying and Spark Plasma Sintering

R. Tavakoli[1]* and M.H. Enayati[2]

Affiliation:

[1] Department of Materials Engineering, Naghshe-Jahan Isfahan Institute of Higher Education, Isfahan, P.O.BOX: 81435-118, Iran

[2] Department of Materials Engineering, Isfahan University of Technology, Isfahan 84156-83111, Iran

***Corresponding Author:** R. Tavakoli, Department of Materials Engineering, Naghshe-Jahan Isfahan Institute of Higher Education, Isfahan, P.O.BOX: 81435-118, Iran*

***Received:** April 15, 2021 **Published:** May 17, 2021*

Abstract:

The purpose of this study is to find out the intermetallic composition of Ni-Cr-Si nanostructures and investigate their formation mechanism during mechanical alloying (MA) and spark plasma sintering (SPS). In this study, Ni (50 wt%), Cr (25 wt%), and Si (25 wt%) were mixed and mechanically alloyed by a planetary ball mill. X-ray diffraction (XRD) test was used to examine the structural changes of the powder particles during mechanical alloying. The morphology and the microstructure of the powder particles were characterized by scanning electron microscopy (SEM). The results showed the formation of two nanocrystalline Ni(Si) and Cr(Si) solid solutions after 70 hours of milling. Then, the resulting powder was sintered by spark plasma sintering (SPS) technique and XRD and SEM were used to examine the structural changes and phase transformations of bulk sample. XRD result showed that three intermetallic compounds ($Ni_{31}Si_{12}$, $Cr_{6.5}Ni_{2.5}Si$, and $Cr_3Ni_5Si_2$) were conducted. Porosity and hardness of the bulk sample were measured. The microhardness value and volumetric porosity of the sintered sample were reported as 973±39.7 kg/mm2 and 8%, respectively.

Keywords: Nanocrystalline, Mechanical Alloying, Ni-Cr-Si, Intermetallic compounds

Introduction

Intermetallic compounds, a class of material composed of definite proportions of two or more elemental metals, are stable within a specific range of chemical composition. Their atomic structure is usually ordered, resulting into a higher melting point and more resistance against deformation at elevated temperatures. High hardness, good wear resistance, and high corrosion and oxidation resistance are their advantages. On the other hand, some of their limitations are low ductility at low temperatures and low creep resistance at high temperatures, restrict their applications in engineering [1-3]. A large number of studies have been conducted in order to improve their ductility, including, the addition of alloying elements, microstructural control, reduction of grain size, and reinforcement of matrix with second phase particles or fibers [4-6].

Despite these limitations, intermetallic compounds are attractive candidates in aerospace, medical engineering, and petrochemical industries [7-8]. Mechanical alloying (MA) has been successfully used for nanocrystalline materials synthesis. The synthesis of nanocrystalline intermetallic compounds using mechanical alloying has been extensively studied [9-10].

One of the most important subsystems in the multicomponent system of the nickel-based alloys is Ni-Cr-Si ternary system [11-12]. Due to the extensive and promising application potentials of the Ni-Cr-Si system, comprehensive information about the phase equilibrium of this system is of great importance since it serves as a map in developing new materials from the system [13-14].

In [15], the structure and properties of Ni-Al-Si nanocrystalline intermetallic compounds produced by mechanical alloying were investigated. The initial components were mechanically alloyed in a planetary ball mill for 50 h. As a result, $Al_{75}Si_{15}Ni_{10}$, $Al_{70}Si_{20}Ni_{10}$, $Al_{65}Si_{25}Ni_{10}$ intermetallic compounds were synthesized. The researchers [16] investigated the effect of 10% of the Chrome as a microstructure enhancer and mechanical properties of Ni_3Al-Cr alloy by mechanical alloying followed by hot pressing at 1250°C under pressure of 150 MPa. They found that the worm particles were dissolved in a hot pressing environment at a temperature of 1250°C to 20%. In addition, the addition of the Chrome increases the stiffness and tensile strength at room temperature, which occurs due to the formation of a strong bond between the Cr and the matrix. In [17], the mechanical alloying of Ni (50 wt.%) and Al (50 wt.%) was investigated. A gradual formation of NiAl nanocrystalline intermetallic compounds with the size of 11 nm was reported. Mechanical alloying of Ni (25 wt.%), Cr (25 wt%), and Al (50 wt%) led to the formation of a Ni-Cr-Al layered structure, which was transformed to Al(Ni-Cr) solid solution as the process continued.

In aim of this study is synthesis of Ni50Cr25Si25 intermetallic compound by mechanical alloying and spark plasma sintering methods and evaluation of microhardness and porosity.

Experimental Procedure

The initial powders used in this study were Ni, Cr, and Si with characteristics presented in Table 1. First, powders of Ni (50 wt.%), Cr (25 wt.%), and Si (25 wt.%) were mixed and mechanically alloyed. The sampling process was carried out after 0, 5, 10, 15, 20, 25, 30, 40, 50, 60, 70 h of mechanical alloying. Structural changes and phase transformations of the powders after 0, 20, and 70 h were examined by XRD and SEM. Mechanical alloying was performed in a planetary ball mill. The body of its chamber was made of a hardened steel containing 500 ml of chromium and its balls were made of a hardened high-carbon steel with a diameter of 20 mm. The rotation speed was 250 rpm, and argon was used to prevent the oxidation of powder particles and the formation of undesirable phases.

Table 1. Particle size, purity, and source of the raw materials

Element	Ni	Cr	Si
Particle size (μm)	25-75	35-62	45
Purity (%)	99.9	99.9	99.9
Source	Russia	Korea	Germany

The powder milled for 70h was sintered by a SPS process according to the parameters shown in Table 2.

Table 2. Sintering parameters for the powder mechanically alloyed for 70 h.

No.	Temp. (0C)	Time (min)	Pressure (MPa)
No. 1	1000	6	50
No. 2	1000	10	50
No. 3	1000	15	50

The variation of temperature vs. time is shown in Figure 1. As seen, the sample was kept at 1000 °C for 6-15 min and the constant pressure of 50 MPa and the whole process took 2500s.

In SPS process, plasma is generated by applying a DC pulse current of 2600A and pressure concurrently. The mold was made of graphite and the size of the samples was 0.5 mm in thickness and 2 mm in diameter. The best sample with the least porosity selected and microhardness test was carried out at the ambient temperature.

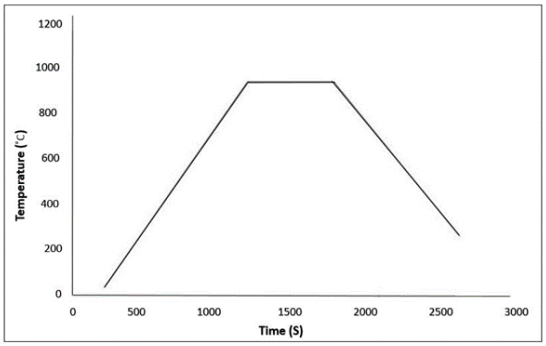

Fig. 1. *The variation of temperature vs. time in SPS process.*

The bulk sample was ground and polished, and the macro hardness test was performed with the force of 30 Kg and the average of eight points was reported as the final value.

Results & Discussions

Investigation of structural changes of MA powder

Figure 2 shows the XRD patterns of Ni50Cr25Si25(%wt.) alloy after 0, 20, and 70 h of mechanical alloying. The XRD pattern of the as-received powder (0h) shows diffraction peaks of the pure crystalline Ni, Cr and Si. With increasing milling time till 20 h, the intensity of Si peaks decreased. After 70h, the Si peaks completely disappeared, and the intensity of Ni and Cr peaks decreased. The width of crystalline Ni and Cr peaks increased with increasing milling time as a result of reduction of grain size and enhancement of internal strain.

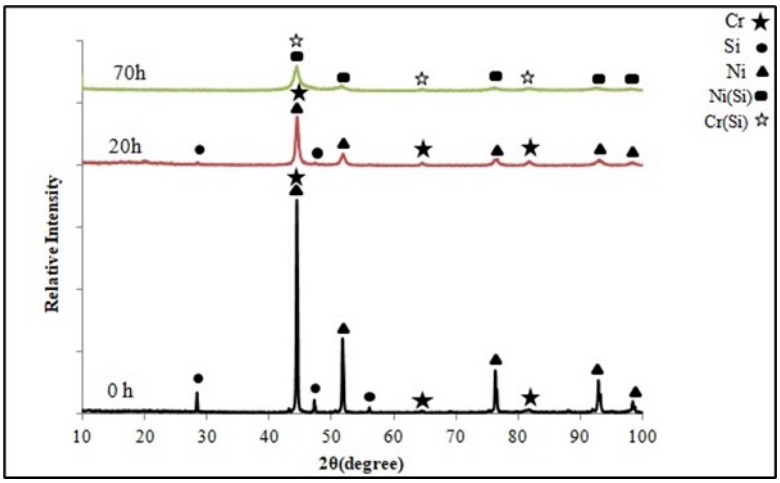

Fig. 2. *XRD patterns of Ni50Cr25Si25(%wt.) alloy after 0, 20, and 70 h of mechanical alloying*

Also, the position of XRD peaks has changed during milling. Basically, this change in peak positions reflects the change in lattice parameter of crystalline phase due to the interstitial and/or substitutional diffusion of different alloying elements in the crystal lattice. Diffusion process during MA is aided by increasing number of lattice defects, formation of nanocrystalline structure and local increase in temperature of milling media.

Morphological images of powder particles after 70h of milling time are shown in Figure 3. With increasing the mechanical alloying time to 70 h, the average size of powder particles reduced and the particle become spherical. This is due to the several simultaneous fragmentation of powder particles, cold welding and work hardening during mechanical alloying. The average size of powder particles after 70 h was 5 μm.

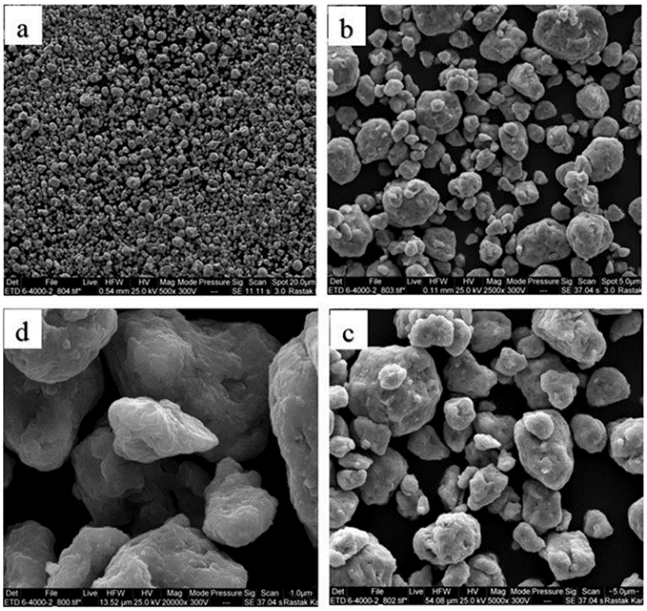

Fig. 3. *SEM images showing morphology of powder particles after 70h of MA:*

Structural investigation of sintered powder

Figure 4 shows the XRD pattern of the sintered $Ni_{50}Cr_{25}Si_{25}$ alloy (No.2). Since SPS was conducted at a high temperature, internal stresses were released and the grains grew which in turn increased the XRD peaks intensity and reduced their width. As seen, holding the powder at 1000°C for 10min led to the formation of $Cr_{6.5}Ni_{2.5}Si$, $Cr_3Ni_5Si_2$ and $Ni_{31}Si_{12}$ intermetallic compounds whereas these compounds were not present in the powder mechanically alloyed for 70 h.

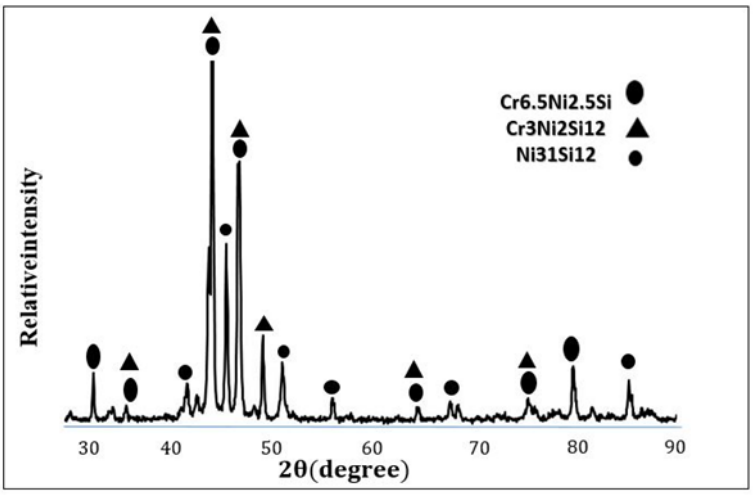

Fig. 4. *XRD pattern of the sintered Ni50Cr25Si25 alloy (sample No. 2).*

Figure 5 shows the SEM image of sintered Ni50Cr25Si25 alloy (No.2). It can be concluded that the microstructure has a good homogeneity and sintering has been significantly completed in a way that the boundary between the powder particles cannot be seen.

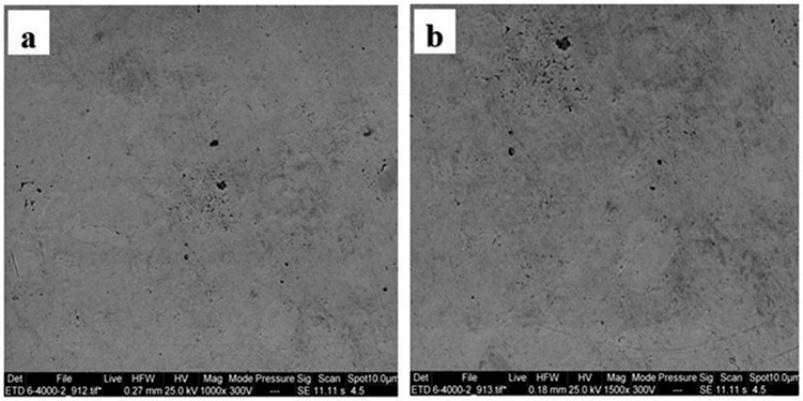

Fig. 5. *SEM image of sintered Ni50Cr25Si25 alloy (sample No. 2).*

To measure the density of sintered Ni50Cr25Si25 alloy, the Archimedes method was used as per B962-14 standard [18]. In this method, first the fully-dried sample is weighted in air (W, actual weight), then it is weighted in pure water (W', apparent weight). Actual density is calculated by Eq. (1)

$$\rho = \frac{\rho' W}{(W - W')} \qquad (1)$$

Where ρ is the actual density, ρ' is density of pure water (1 gr/cm3), W is the mass of sample in the air (actual weight), and W' is the mass of sample in a fluid (apparent weight). Porosity percentage was calculated by Eq. (2).

$$\%P \quad \frac{\text{Saturated weight—dried weight}}{\text{Saturated weight— immersed weight}} \qquad (2)$$

The value of porosity for samples of No. 1, No.2 and No. 3 was obtained 11.45, 8 and 9%, respectively. It means the optimum condition of sintering process is sintering condition of No. 2.

The hardness of sintered Ni50Cr25Si25 alloy (No. 2) was determined by microhardness test. The average hardness of the sample is 973±39.7 kg/mm2. Other studies [19-21] on evaluation of microhardness of intermetallic compounds have shown the high hardness of intermetallic compounds.

Conclusion

1-Ball milling of Ni (50 wt.%), Cr (25 wt.%), and Si (25%) powders led to the formation of Ni(Si) and Cr(Si) solid solutions.
2- Sintering treatment via SPS process at 1000 0C for 10 minutes led to the formation of a bulk sample with relative density of 92%
3-XRD results of the bulk sample showed that SPS led to the formation of Cr6.5Ni2.5Si, Cr3Ni5Si2 and Ni31Si12 intermetallic compounds.
4-The microstructure of SPS sample showed a good homogeneity.
5-SEM of sintered sample showed that the sintering treatment was fully done and the boundary between the powder particles could not be seen.
6-The sintered sample has a high hardness of about 973±39.7 kg/mm2.

Conflict of Interest

The authors declare no conflict of interest.

Acknowledgement

The authors gratefully acknowledge Mr. M.H. Enayati for supporting the production of samples and mechanical material also Materials Engineering laboratory, Naghshe-Jahan Isfahan Institute of Higher Education, Isfahan, Iran

References

1. M. Atzmon, Characterization of AlNi formed by a self-sustaining reaction during mechanical alloying. Mat. Sci. and Eng., 134(1991), p.1326.
2. Y. Li, J. Guo, H. Ye, Zirconium-Induced Softening in Hyperstoichiometric Ni3Al, J. Mater. Sci. Technol., 21(2005), p.207.
3. S.N. Hosseini, T. Mousavi, F. Karimzadeh, M.H. Enayati, Thermodynamic aspects of nanostructured CoAl intermetallic compound during mechanical alloying., J. Mater. Sci. Technol., 27(2011), p.601.
4. S.Z. Anvari, F. Karimzadeh, M.H. Enayati, Synthesis and Characterization of NiAl-Al2O3 Nanocomposite Powder by Mechanical Alloying. J. Alloy. Compd., 477(2009), p.178.
5. L. Sheng, W. Zhang, J. Guo, F. Yang, Y. Liang, H. Ye, Effect of au addition on the microstructure and mechanical properties of NiAl intermetallic compound, Intermetallics 18(2010), p.740.
6. J.T. Guo, L.Y. Sheng, Y. Xie, Z.X. Zhang, V.E. Ovcharenko, H.Q. Ye, Microstructure and mechanical properties of Ni3Al and Ni3Al–1B alloys fabricated by SHS/HE, Intermetallics, 19(2011), p.137.
7. N.S. Stoloff, T. Liu, S.C. Deevi, Emerging Applications of Intermetallics, Intermetallics, 8(2000), p.1313.
8. S.S. Cohen, P.A. Piacente, G. Gildenblat G, D.M. Brown, Platinum silicide ohmic contacts to shallow junctions in silicon, J. Appl. Phy., 53(1982), p.8856.
9. C. Suryanarayana, Mechanical alloying and milling, Pro. Mater. Sci., 46(2001), p.1.
10. Y. Yu, J. Zhou, J. Chen, H. Zhou, C. Guo, B. Guo, Synthesis of nanocrystalline Ni3Al by mechanical alloying and its microstructural characterization, J. Alloy. Compd., 498(2010), p.107.
11. S.K. Tung, L.C. Lim, M.O. Lai, Solidification phenomena in nickel base brazes containing boron and silicon, Scr. Mater., 34(1996) 763.
12. J.S.C. Jang, H.P. Shih, Evolution of microstructure of AISI 304 stainless steel joint brazed by mechanically alloyed nickel base filler with different silicon content, J. Mater. Sci Lett., 22(2003), p.79.
13. G. Ceccone, M.G. Nicholas, S.D. Peteves, A.A. Kodentsov, J.K. Kivilahti, F.J.J. Van Loo, The Brazing of Si3N4 with Ni-Cr-Si Alloys, J. Eur Cera. Soc., 15(1995), p.563.
14. J.R. McDermid, R.A. Drew, Thermodynamic brazing alloy design for joining silicon carbide, J. Am. Ceram. Soc., 74 (1991), p.1855.
15. M. Jain, S.P. Gupta, Formation of intermetallic compounds in the Ni–Al–Si ternary system, Mater Charact., 51(2003), p.243.
16. J. Garcia Barriocanal, G. Pérez, G. Garcés, P. Adeva, Microstructure and mechanical properties of Ni3Al base alloy reinforced with Cr particles produced by powder metallurgy, Intermetallics, 14(2006), p.456.

17. A.S. Bidabadi, M.H. Enayati, E. Dastanpoor, R.A. Varin, M. Biglari, Nanocrystalline intermetallic compounds in the Ni–Al–Cr system synthesized by mechanical alloying and their thermodynamic analysis, J. Alloy. Compd., 581(2013), p.91.

18. Standard Test Methods for Density of Compacted or Sintered Powder Metallurgy (PM) Products using Archimedes' Principle. ASTM. 2008.

19. Jafari, A 2014 MS Thesis (Semnan Univerity).

20. S. Harsha, D.K. Dwivedi, A. Agarwal, Influence of CrC addition in Ni-Cr-Si-B flame sprayed coatings on microstructure, microhardness and wear behaviour, Int. J. Adv. Manuf. Technol. 38(2008), 93.

21. M. Abbasi Baharanchi, F. Karimzadeh, M.H. Enayati, Effects of friction stir process parameters on microstructure and mechanical properties of aluminum powder metallurgy parts, J. Adv. Mater. Processes 4(2016) 38

Citation: Tavakoli R, Enayati M.H "Synthesis of Intermetallic Compounds in the Ni-Cr-Si System by Mechanical Alloying and Spark Plasma Sintering". SVOA Materials Science &Technology, 2021, 3(3) Pages: 45-50.