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Effects of the temperature of hot isostatic pressing treatment on Cr–Si targets

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Abstract

Commercial as-hp (hot pressing) treated Cr–Si targets are used throughout this study, with three different compositions: Cr20–Si80, Cr35–Si65 and Cr50–Si50. To evaluate the effects of microstructure and properties of as-hp treated Cr–Si targets by hot isostatic pressing (HIP) SEM, XRD and porosity inspections were performed. The experimental results showed that the 1373 K, 1750 MPa, 4 h HIP treated with three different Cr–Si targets had suppressed porosities successfully. The most efficient was Cr50–Si50 target subjected to HIP treatment. Porosity decreased about 60% after HIP treatment, and both the nitrogen and oxygen concentrations of the targets were slightly increased after HIP treatment. This was especially true for the single silicon in Cr–Si targets such as Cr20–Si80 and Cr35–Si65. The aim of this paper is to discuss these methods and finding suitable temperatures for the HIP for Cr–Si targets.

Keywords: A. Hot pressing; A. Hot isostatic pressing; B. Porosity; Cr–Si targets

1. Introduction

Most targets could be made by casting, because the metals have low melting points and good toughness. However, some activated or brittle elements such as chromium and silicon are not suitable for casting, because they are easily oxidized and cracked during the process. To make the Cr–Si targets in powder metallurgy; it is better to produce them by hot pressing. Hot pressing (HP) is putting the powder in a die and applying force with vertical (or horizontal) pressure heads. The heaters around the die would be turned on in a vacuum or with atmosphere [1–4]. The products made by hot pressing would still be porous or coarse, and the size is limited. For finer and larger targets, we can apply advanced method, like hot isostatic pressing.

Hot isostatic pressing (HIP) is the application of high pressure equally on all sides of an object. HIP is a material pressing technique in which high isostatic pressure is applied to a powder part or compact at elevated temperature to produce particle bonding. This process usually results in the manufacture of a fully dense body. During this process, the compact is subjected to equal pressure from every side [5–7]. HIP is used for dense high performance ceramics, to remove porosity from castings, the consolidation of PM materials and surface coatings, diffusion bonding and improvement of weld integrity [8–12]. As shown in Fig. 1, HIP equipment was from a Flow Autoclave Pressure System, Inc. This HIP equipment provided an uniform rapid cooling (URC) system and a quick heating which result in very short cycle time and high productivity.

In this study, three different HIP temperatures are utilized, including 1323 K, 1373 K and 1423 K. The processing pressure and soaking time for the HIP treatment were 175 MPa and 4 h, respectively. Porosity test, nitrogen and oxygen concentration test, X-ray diffraction analysis and microstructure inspection were used to evaluate the effects of HIP treatment at different temperatures for as-hp treated Cr–Si targets.

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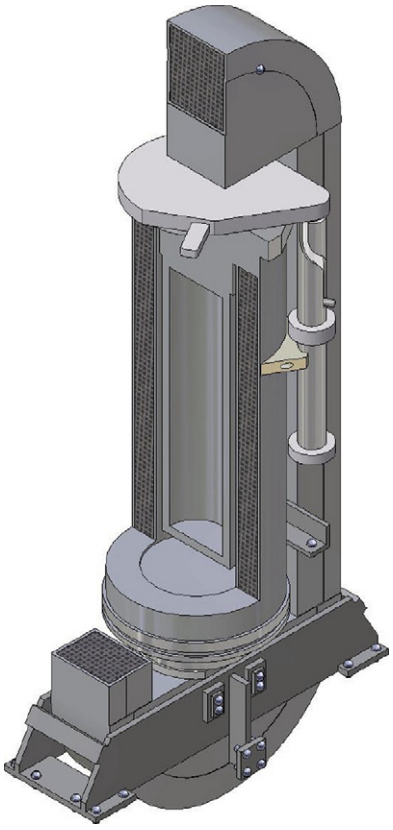

Fig. 1. Schematic diagram of HIP equipment.

2. Experimental

Both chromium and silicon are rapidly oxidized, and powder metallurgy is usually used to make targets out of such materials, especially in hot pressing. To achieve better quality targets, we should use some advanced methods, such as hot isostatic pressing. HIP involves the application of high pressures and temperatures through the medium of a pressurized gas, such as argon or nitrogen, to remove internal pores and voids, thus increasing density and upgrading properties. HIP consolidates powders of metals, ceramics, or carbides into fully dense, complex parts with net or near net shapes.

The aim of this paper is to discuss the methods and to find a suitable temperature of HIP for Cr–Si targets. In this study, the HIP pressure was maintained at 175 MPa, the soaking time 4 h and the three different temperatures were 1323 K, 1373 K and 1423 K. Commercial HP treated Cr–Si targets were used throughout this study, and they had three different compositions: Cr20–Si80, Cr35–Si65 and Cr50–Si50. To evaluate the effects of microstructure and properties of the Cr–Si target by HIP process, SEM, XRD and porosity inspections were performed.

3. Results and discussion

3.1. Porosity test

The porosity test followed the ASTM C373 standard. During HIP plastic flow, power law creep, grain boundary sliding in the particles and bulk diffusion in the particle contacts may contribute to the densification [2]. Fig. 2 represents the porosity of Cr–Si targets after HIP treatment at different temperatures. It shows the porosity was significantly reduced before 1423 K HIP treatment. The porosity of Cr50–Si50 target was reduced down to 40% after HIP treatment at 1373 K. The minimum amount of porosity of Cr20–Si80 target was obtained for samples after HIP treatment at 1323 K. When the temperature of HIP was increased from 1373 K to 1423 K, the porosity rapidly increased. Because the silicon exists singly inside the Cr20–Si80 and Cr35–Si65 targets, the new pores would emerge by diffusion and adsorption of silicon elements.

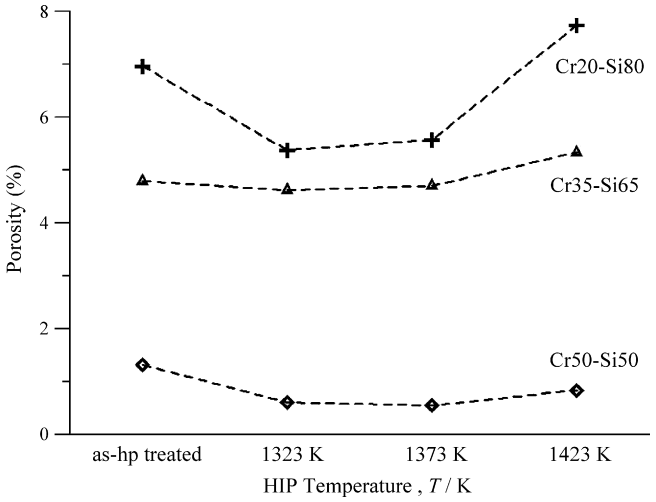

Fig. 2. Comparison of the porosities of as-hp treated and after HIP treatment at different temperatures for different compositions of Cr–Si targets.

3.2. Concentration of nitrogen and oxygen

Figs. 3 and 4 show the concentration of nitrogen and oxygen inside the Cr–Si targets for different compositions after HIP treatment. The results show the silicon element was singly existed in the Cr20–Si80 and Cr35–Si65 targets, and the concentrations of impurities would rise up after HIP treatment. There was still residual air inside the vessel before heating, and

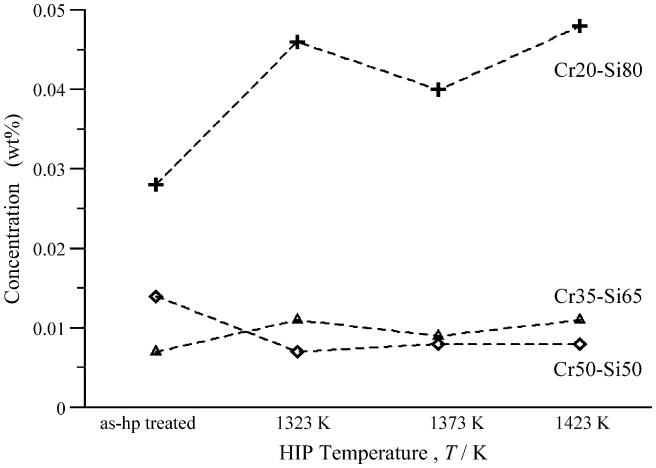

Fig. 3. Comparison of the nitrogen concentration of as-hp treated and after HIP treatment at different temperatures for different compositions of Cr–Si targets.

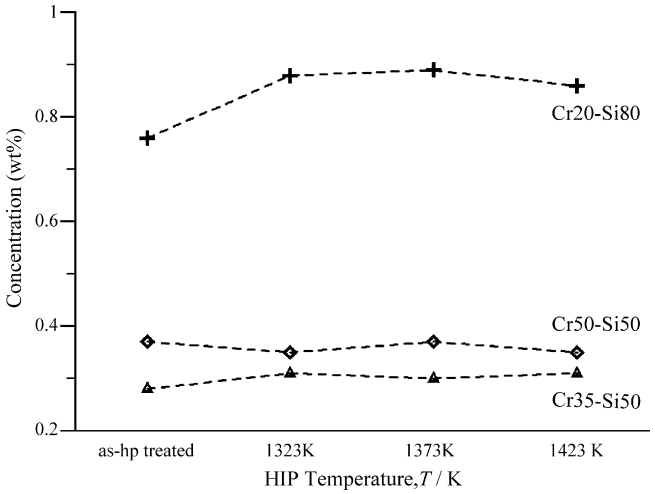

Fig. 4. Comparison of the oxygen concentration of as-hp treated and after HIP treatment at different temperatures for different compositions of Cr–Si targets.

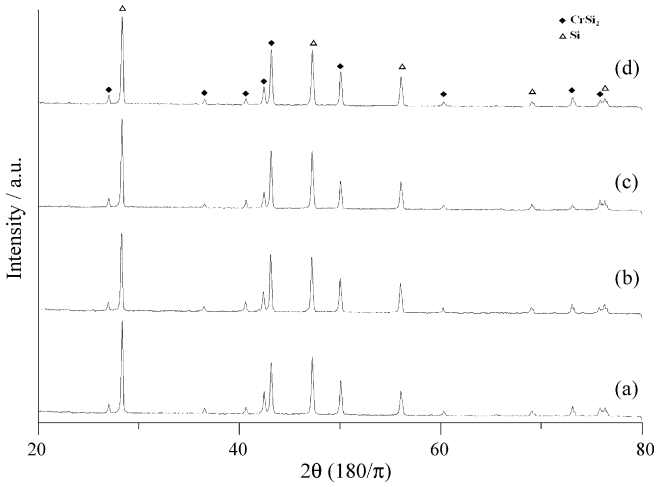

Fig. 6. XRD patterns of as-hp treated and after HIP treatment at different temperatures for Cr35–Si65 target (a) as-hp treated, (b) 1323 K, (c) 1373 K and (d) 1423 K.

it was hard for the vacuum system to completely eliminate the nitrogen and oxygen during the HIP process. Thus SiO_2 and Si_3N_4 would be formed inside of the targets, and the concentrations would be increased. Because CrSi and $CrSi_2$ are stable intermetallic compounds, little SiO_2 and Si_3N_4 would emerge. Both the nitrogen and oxygen concentrations were decreased on the Cr50–Si50 target after HIP treatment. With the reducing porosity, there were fewer and smaller pores to be adsorbed and the concentrations went down.

3.3. XRD analysis

$CrSi_2$ is a polycrystalline of sintered material. The structure of $CrSi_2$ crystals was determined using X-ray diffraction. The tested sample consisted of hexagonal $CrSi_2$ [13–14]. Fig. 5 represents the XRD pattern of as-hp treated and HIP treated at different temperatures Cr20–Si80 target. In the XRD patterns were identified as silicon and $CrSi_2$. A similar XRD pattern was shown in Fig. 6, but there was different result in Fig. 7. Crystal phases of Cr20–Si80 and Cr35–Si65 targets after HIP treatment were silicon and $CrSi_2$ phases; however there were CrSi and $CrSi_2$ phases in the HIP Cr50–Si50 target. The intensity of the $CrSi_2$ phase was raised in various compositions of Cr–Si targets after HIP treatment. This means the crystal structure of $CrSi_2$ was improved after HIP treatment. Making a comparison of the XRD patterns of different composition Cr–Si targets after HIP treatment, this experiment result showed that a decrease in the silicon content of the Cr–Si target could enhance the $CrSi_2$ precipitation.

3.4. SEM observation

Fig. 8 is the secondary electron images (SEI) of as-hp treated and HIP treated at different temperatures Cr20–Si80 targets. Increasing HIP temperature did not reduce the porosity, and the optimal HIP temperature appeared at 1323 K. Figs. 9 and 10 are the SEM of as-hp treated and different temperatures HIP treated

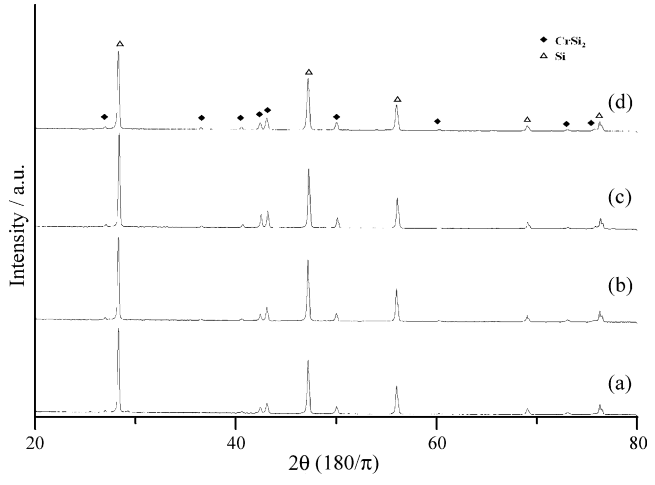

Fig. 5. XRD patterns of as-hp treated and after HIP treatment at different temperatures for Cr20–Si80 target (a) as-hp treated, (b) 1323 K, (c) 1373 K and (d) 1423 K.

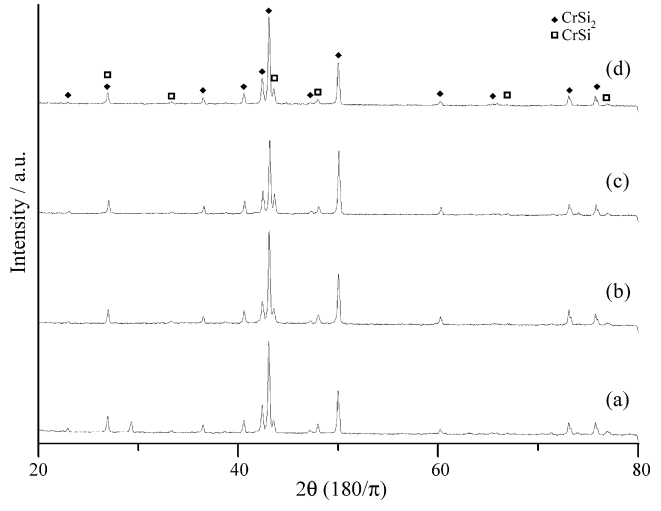

Fig. 7. XRD patterns of as-hp treated and after HIP treatment at different temperatures for Cr50–Si50 target (a) as-hp treated, (b) 1323 K, (c) 1373 K and (d) 1423 K.

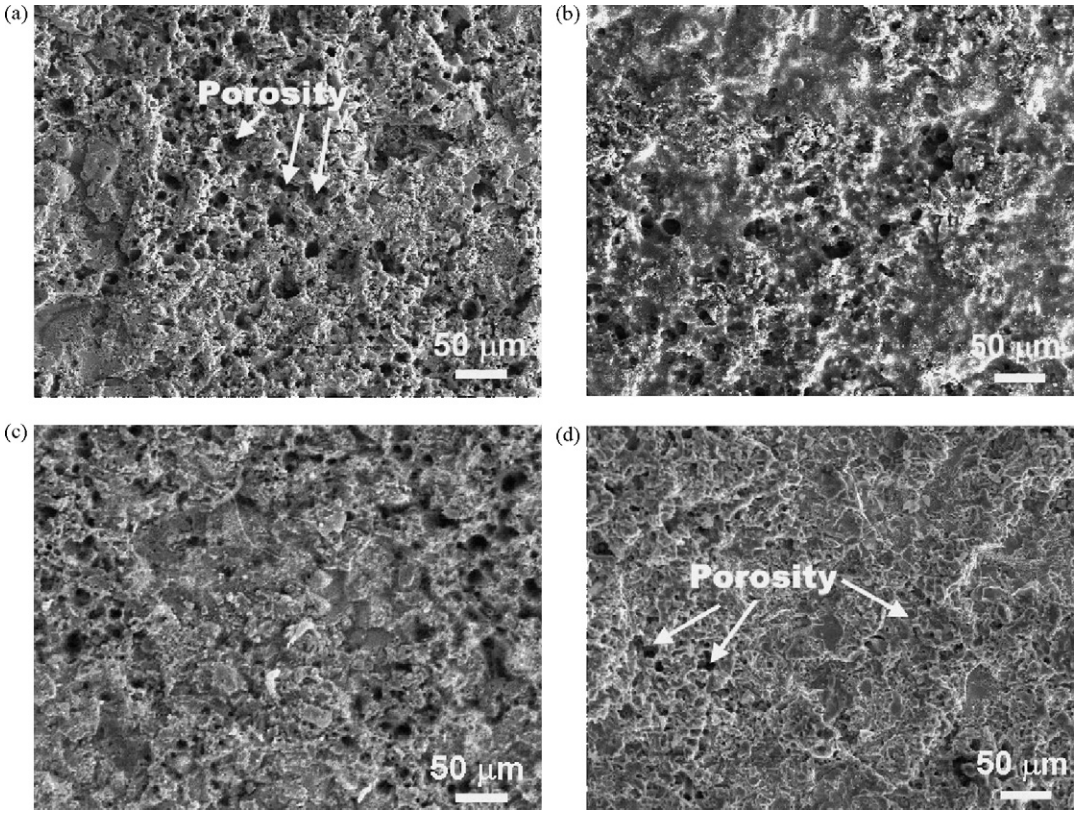

Fig. 8. SEI micrographs of as-hp treated and HIP treatment at different temperatures for Cr20–Si80 target (a) as-hp treated, (b) 1423 K, (c) 1373 K and (d) 1323 K.

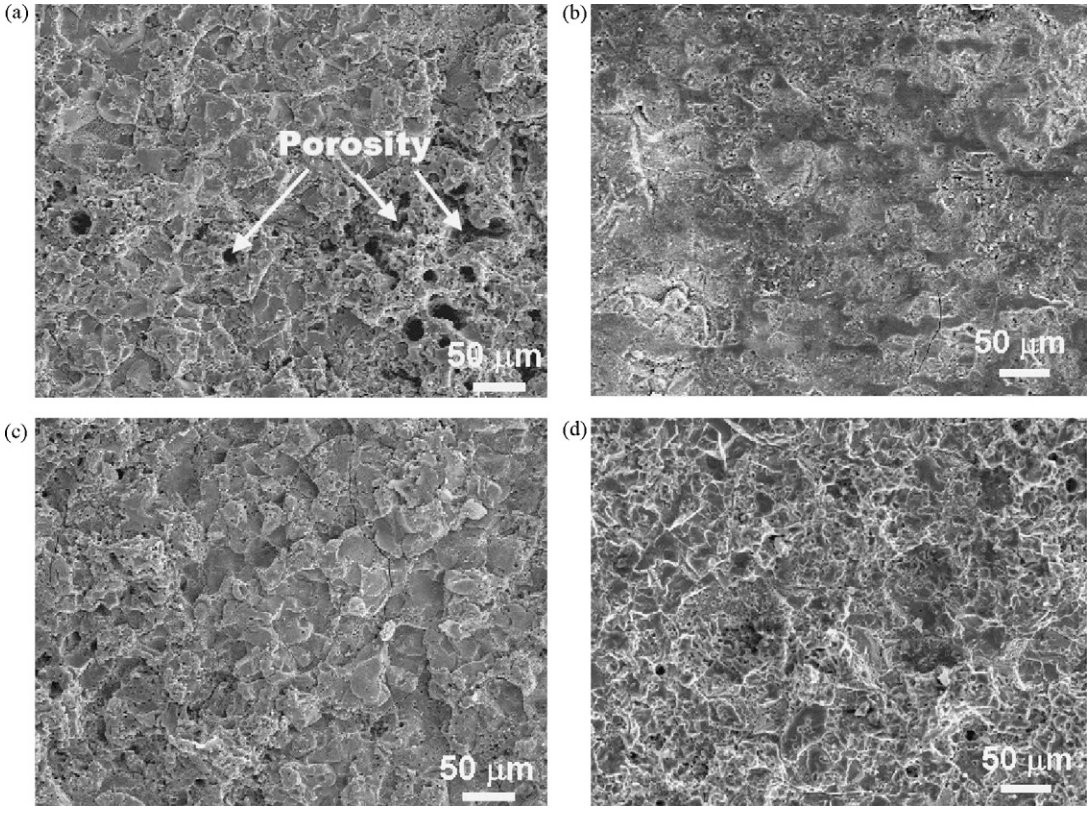

Fig. 9. SEI micrographs of as-hp treated and HIP treatment at different temperatures for Cr35–Si65 target (a) as-hp treated, (b) 1423 K, (c) 1373 K and (d) 1323 K.

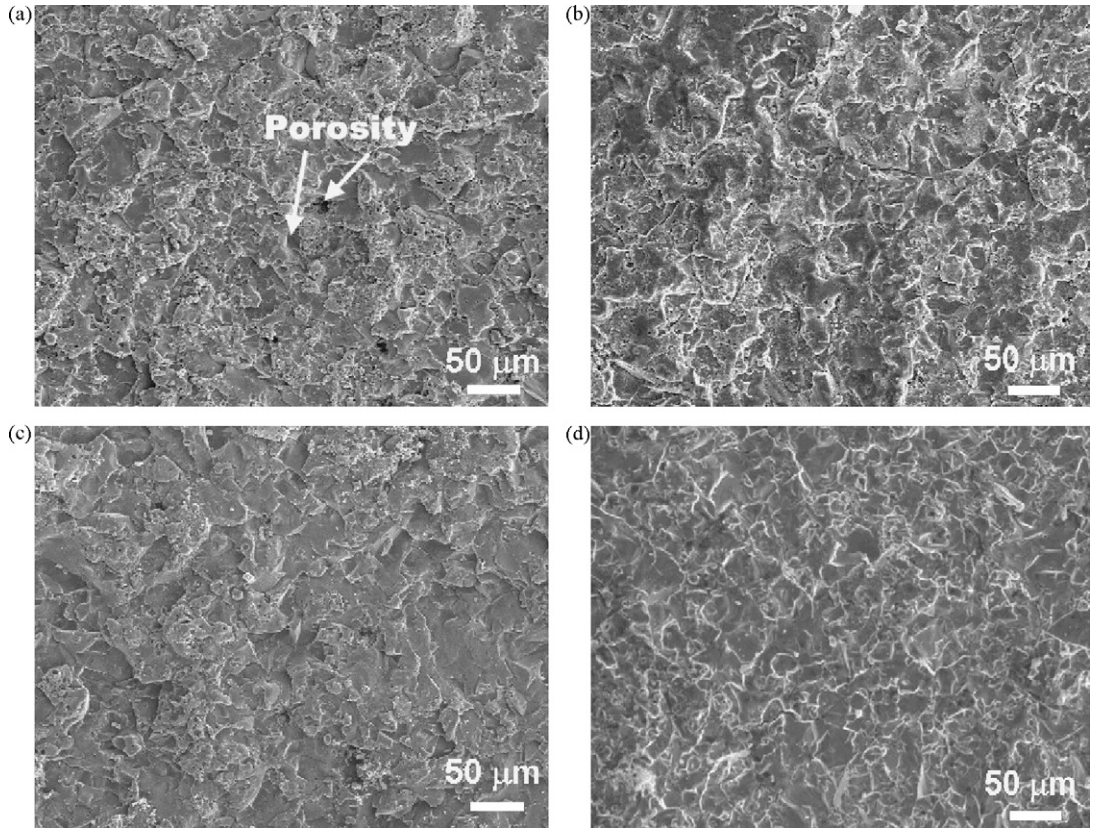

Fig. 10. SEI micrographs of as-hp treated and HIP treatment at different temperatures for Cr50–Si50 target (a) as-hp treated, (b) 1423 K, (c) 1373 K and (d) 1323 K.

Cr35–Si65 and Cr50–Si50 targets. HIP treatment clearly eliminated and decreased the porosities, and the optimal HIP temperature appeared at 1373 K. When the HIP temperature increased to 1423 K, the bonding force of the chromium and silicon elements was weak, especially for the Cr20–Si80 target. That shows an increase in the amount of silicon, would cause a poor microstructure to exist in the Cr–Si targets after HIP treatment. Fig. 10 shows the Cr50–Si50 target could show a better microstructure and porosity after HIP treated at different temperatures.

Fig. 11 shows the backscattered electron images (BEI) of as-hp treated and 1373 K, 175 MPa, 4 h HIP treatment for the Cr50–Si50 target. In the images, the white area is CrSi and gray area is $CrSi_2$. Fig. 11(a) shows the compounds of CrSi, $CrSi_2$ and Cr_5Si_3 existed in the as-hp Cr50–Si50 target, and Fig. 11(b) shows that much of the dense compounds of CrSi and $CrSi_2$ would exist in the Cr50–Si50 target after HIP treatment. They would more efficiently reduce the porosity and improve the microstructure. Fig. 12 is the EDS results of CrSi and $CrSi_2$ for the Cr50–Si50 target after 1373 K, 175 MPa, 4 h HIP treatment. The structure of

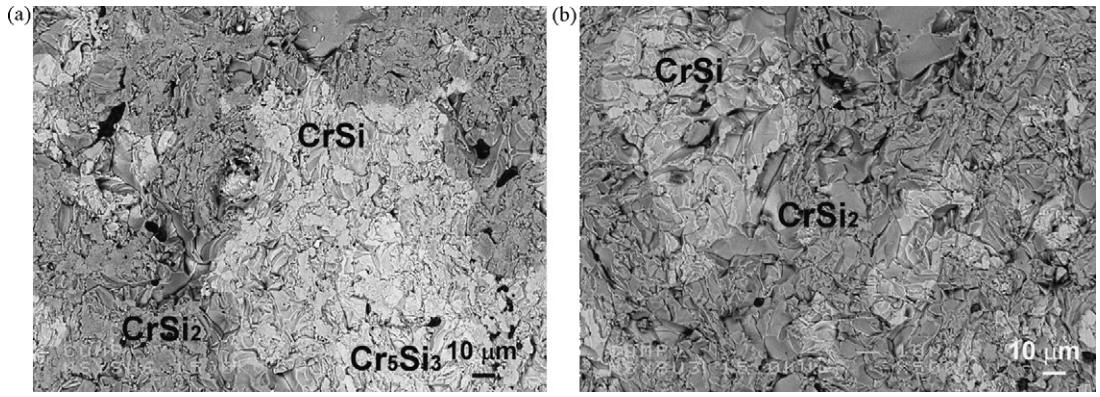

Fig. 11. BEI micrographs of (a) as-hp treated and (b) 1373 K, 175 MPa, 4 h HIP treatment for Cr50–Si50 target.

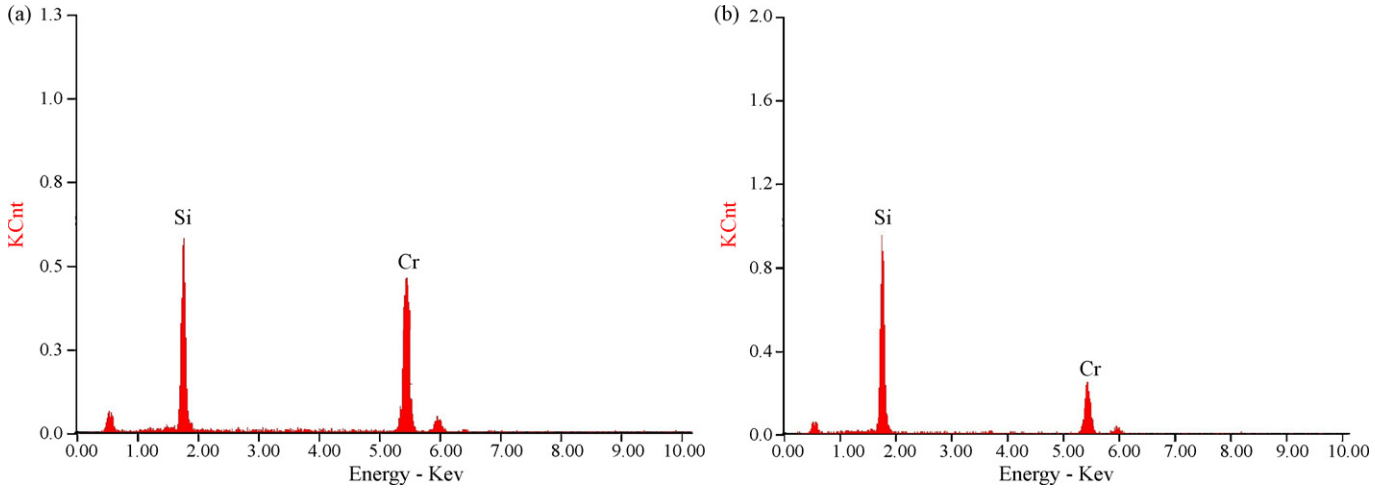

Fig. 12. EDS results of (a) CrSi and (b) $CrSi_2$ for Cr50–Si50 target after 1373 K, 175 MPa, 4 h HIP treatment.

CrSi and $CrSi_2$ was apparent after HIP treatment, and there was no more Cr_5Si_3 existed in the Cr50–Si50 target.

4. Conclusion

(1) The experimental results show that 1373 K, 1750 MPa, 4 h of HIP treatment for three different Cr–Si targets was optimal. The most efficient was Cr50–Si50 produced by optimal HIP treatment, which decreased to 40% in porosity after HIP treatment.
(2) The higher silicon content of the Cr–Si target resulted in increased porosities, due to the new porosity produced by diffusion of silicon at 1423 K HIP treatment.
(3) Nitrogen and oxygen concentrations of the Cr–Si target were increased after HIP treatment, created by the refined gas in the close vessel of the HIP equipment.

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