Research Article

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Microstructure and properties of Co–Al porous intermetallics fabricated by thermal explosion reaction

<https://doi.org/10.1515/htmp-2020-0076>

received November 01, 2017; accepted April 26, 2018

Keywords: porous materials, intermetallics, powder metallurgy, thermal explosion

Abstract: Co–Al porous intermetallics were fabricated by an efficient and energy-saving method of thermal explosion (TE) reactions. The effects of Co/Al molar ratios on the temperature profiles, phase compositions, expansion behaviors, density, pore characteristics, and oxidation resistance were investigated. When the target furnace temperature was set at 700°C, there was an obvious exothermic peak in the temperature profiles. The ignition temperatures were in the range of 600–645°C, and the combustion temperatures were in the range of 984–1,421°C. Co–Al porous intermetallics had the open porosity of 27–43%, and the pores were from nonfully dense green compacts and explosion behaviors of TE. The specimen with Co:Al = 2:9 possessed a higher open porosity of 42.8%, the lowest density of 1.86 g cm⁻³, and the largest volume expansion of 76.7%. The porous specimens with Co:Al = 1:1 possessed the highest open porosity of 43.2%, the lowest volume expansion of 12.3%, and the highest density of 3.42 g cm⁻³. All Co–Al porous intermetallics showed excellent oxidation resistance at 650°C in air, especially the specimen with Co:Al = 1:1 had the highest oxidation resistance.

1 Introduction

Porous materials have been widely investigated attributed to their low density, large specific surface area, soundproofed, insulation, good permeability, and high specific strength [1–3]. Porous materials are divided into three classes according to the International Union of Pure and Applied Chemistry classification: microporous, mesoporous, and macroporous [4,5]. Microporous materials are composed of a solid base and interconnected pores <2 nm, which are widely used in the applications of heterogeneous catalysis, adsorption, and gas storage [6]. Mesoporous materials have a distribution of pores in the range of 2–50 nm, which are widely applied in the fields of chromatography, catalysis, and electrochemistry [4,7]. Macroporous materials have interconnected pores >50 nm, which include organic and inorganic porous materials.

Organic porous materials are only used in the fields of water treatment and biology due to their poor mechanical properties and thermal stabilities at high temperatures and high pressure, negligible resistance to environmental corrosion and organic solvents. Ceramic porous materials belong to inorganic porous materials and possess inherent advantages of high melting point, excellent corrosion and wear resistance, low thermal mass, and thermal conductivity [8,9], allowing for their use in molten metal filtration, thermal insulation, and bio-scaffolds for tissue engineering [10–12]. However, the brittleness, low thermal shock resistance, and poor weldability have limited their applications. Metal porous materials have advantages over ceramics, such as high mechanical strength, good thermal shock resistance, weldability, and excellent machining property, but their strength is low, and oxidation and corrosion resistance

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are poor at high temperatures. These limit their applications in high temperature and corrosion environment [13].

The intermetallic compound is a new type of inorganic material between metal and ceramic materials. In this material, most of the element bind by the covalent bond and a few elements bind by the metal bond; hence, it has the common advantages of both metal and ceramic materials [14–18]. The porous intermetallic compound has many excellent properties, such as low density, high elastic modulus, improved specific strength and stiffness, excellent oxidation resistance, good corrosion resistance, and machinability. Therefore, it has wide application prospects as a substitute material of stainless steel and catalyst support material at high temperatures [18,19].

Recently, Al-based porous intermetallics have been widely investigated for their use in catalysts, filters, and heat insulation components in special environments [20–22]. Numerous studies of Al-based porous intermetallics are mainly focused on Ti–Al (Ti_3Al , TiAl , and TiAl_3) [23,24], Fe–Al (Fe_3Al and FeAl) [25–27], and Ni–Al (Ni_3Al and NiAl) [28], especially the research and application of Ti–Al and Fe–Al intermetallics have made great progress. However, the report of Co–Al porous intermetallics is rare. Yeh prepared Co–Al intermetallic compound by using a self-propagating high-temperature synthesis (SHS) method but did not emphasize it as porous material [29]. SHS is an autogenous process and derives its energy from the exothermic reactions of the reactants [30]. It is used to prepare porous materials, but easy to form structural defects, such as deformation, lamination, and cracks [31–33]. A thermal explosion (TE) is an efficient and energy-saving method for preparing porous materials. It can avoid the deformation and cracks during the sintering process because all the reactants are heated simultaneously in the furnace during the TE reaction [5].

In this study, Co–Al porous intermetallics were fabricated by the TE reaction. TE curves under different Co/Al molar ratios were measured. The effects of Co/Al molar ratios on the temperature profiles, phase compositions, expansion behaviors, density, pore characteristics, and oxidation resistance were investigated.

2 Experimental

Cobalt powders (Co, $\sim 18\ \mu\text{m}$, 99.9% purity) and aluminum powders (Al, $\sim 47\ \mu\text{m}$, 99.0% purity) were mixed with five different molar ratios, including Co:Al = 1:1, 2:5,

1:3, 4:13, and 2:9, which correspond with Al contents of 31.40, 53.4, 58.0, 59.8 and 67.3 wt%, respectively. The mixtures were blended in a planetary ball mill (QM-ISP 2CL) at 450 rpm for 4 h using agate balls (Φ 2, 4, 6, and 10 mm) in agate vessels (inner diameter 72 mm, outer diameter 92 mm, and height 94 mm) at room temperature. The weight ratio of agate balls to powders was 1:1. Ethanol was added as the milling medium during blending for mixing more uniformity and avoiding the oxidation. The mixed powders were dried completely in an oven at 40°C for 24 h.

The mixture was uniaxially pressed at a pressure of 200 MPa in a stainless steel cylindrical mold to form a disc green compact with a diameter of 16 mm and a thickness of $\sim 3\ \text{mm}$ at room temperature. The green compacts were then sintered in a vacuum tubular furnace (OTF-1200X) under an atmosphere of argon. The target furnace temperature was set at 700°C for 30 min and achieved with a constant rate of 10°C/min, and then the specimens were obtained after cooling with the furnace to the room temperature. To confirm the existence of the TE reaction, a couple of thermocouples (WRe3–WRe25) with a diameter of 0.1 mm were placed between two similar specimens to record the actual temperature of the specimen.

The dimensions of the specimens were measured before and after sintering to characterize the expansion behavior, including the expansions in volume, and in the axial and radial directions. The density of the porous intermetallics was measured with the Archimedes method. The open porosity (θ) was calculated using the following equation:

$$\theta = \frac{(M_2 - M_1)\rho_2}{(M_3 - M_1)\rho_1} \% \quad (1)$$

where M_1 is the mass of the dried porous materials in air, M_2 is the mass of porous materials filled with oil in the air, M_3 is the mass of porous materials filled with oil in water, ρ_1 is the density of oil, and ρ_2 is the density of water.

Sintered product compositions were determined by X-ray diffraction (XRD, Bruker D8ADVANCE) using Cu target ($\lambda = 0.15406\ \text{nm}$) with a radiation of 40 kV and 150 mA settings. The optical metallography of polished specimens was observed on an optical microscope (OPTPro3000). Scanning electron microscopy (SEM, Quanta 250) equipped with energy dispersive spectrometer (EDS, UANTAX400) was performed to examine the microstructure and elemental composition of Co–Al porous intermetallic fractures. The oxidation experiments were conducted at 650°C for 96 h at air atmosphere. The oxidation kinetics of Co–Al porous

intermetallics was determined by measuring the change in mass every 24 h as a function of the exposure time.

3 Results and discussion

3.1 Characteristics of the TE reaction

Figure 1 shows the TE profiles and the partial magnified exothermic peaks of the specimens with different Co/Al molar ratios. The ignition temperature (T_{ig}) and the combustion temperature (T_c) marked in Figure 1 were two important parameters of the TE process. TE curve was divided into three parts by T_{ig} and T_c . In the first part from room temperature to T_{ig} , the temperature increased slowly according to the furnace setting. The second part was from T_{ig} to T_c ; in this part, the temperature increased sharply when the specimen was heated to T_{ig} , implying that there was a reaction between Co and Al and the reaction was violent. Large amounts of heat released from the intermetallics formation in the Co–Al system and led to an instant rise in the temperature–time profiles. Finally, the temperature quickly cooled to the furnace setting temperature (700°C) and kept balance for 30 min. TE compact was obtained after cooling with the furnace to room temperature. The values of T_{ig} shown in Figure 1a–e were 645.1, 600.0, 609.1, 618.0, and 626.9°C. The values of T_{ig} increased with increasing Al content which means that the specimen was more difficult to be ignited (Figure 1b–e). The formation of Co–Al intermetallics required the temperature rising to a certain extent in the TE reaction. The maximum value of T_{ig} was 645.1°C and neared the melting temperature of Al (660°C), which implied that the TE reaction started from solid Co–solid Al. The values of T_c shown in Figure 1a–e were 1327.2, 986.4, 1156.3, 1067.1, and 1420.9°C, which were higher than the furnace temperature (700°C) and the melting temperature of Al (660°C), indicating there was an obvious heat release in the Co–Al TE process and the Co–Al TE reaction mainly occurred in solid Co–liquid Al [5,34]. The values of T_c were different with different Co/Al molar ratios because there were different Co–Al TE reactions, sintered products (XRD result in Figure 2), and release heat.

3.2 Phase transformation

Figure 2 shows the XRD spectra of TE products with different Co/Al molar ratios. XRD results indicated that

only the specimen with Co:Al = 1:1 produced a single-CoAl phase. For the other four compositions investigated (Co:Al = 2:5, 1:3, 4:13, and 2:9), the products contain, as the most conspicuous phase, the intermetallics having the near stoichiometry of the green mixture together with small amounts of CoAl (Co:Al = 2:5, 4:13, and 2:9) and unreacted Al (Co:Al = 2:9), Al_2O_3 (Co:Al = 1:3, 4:13, and 2:9). The reason to explain this phenomenon was that only CoAl melted congruently at 1,645°C, while Co_2Al_5 , Co_4Al_{13} , and Co_2Al_9 decomposed according to peritectic reactions at the temperature ranging between 943 and 1,170°C [35]. The intermetallics of the specimen with different Co/Al molar ratios were mainly CoAl, Co_2Al_5 , Co_4Al_{13} , and Co_2Al_9 corresponding to their near Co/Al molar ratio.

3.3 Expansion behavior and density

After the TE reaction, specimens with different Co/Al molar ratios had various macroscopic volume changes, as shown in Figure 3a. All of the sintered products maintained the original cylindrical shape and did not crack, but some specimens (Co:Al = 2:5 and 1:3) appeared slightly deformation or defects because of the violent TE reaction. Additionally, Figure 3b shows the expansion characteristics as a function of the Al content. The volume expansion was proportional to the dimension in the axial direction and exponentially to the dimension in the radial direction. The dimension of the specimens with Co:Al = 1:1 and 4:13 increased 0.5% and 7.5% in the radial direction and 11.2% and 39.25% in the axial direction, respectively; hence, the specimens basically kept the original size in the radial direction, and the volume expansion ratios were 12.3% and 39.3%, respectively. The dimension of the specimens with Co:Al = 2:5 and 1:3 decreased 8.1% and 4.6% in the radial direction and increased 70.2% and 31.7% in the axial direction, respectively; hence, the specimen shrank in the radial direction and became thicker when compared with the green compact, and the volume expansion ratios were 43.8% and 19.7%, respectively. The dimension the specimen with Co:Al = 2:9 increased 18.3% in the radial direction and 26.2% in the axial direction; hence, the specimen expanded in both radial and axial directions, and the volume expansion ratio was 76.7%. The difference expansion ratios of different Co/Al molar ratios were due to the different TE reactions as well as the exothermic action. The specimen with Co:Al = 2:9 showed noticeable volume expansion because the value of T_c was the highest among all specimens (Figure 1e).

Figure 1: Temperature–time profiles (left) and magnified exothermic curves (right) with different Co/Al molar ratios: (a) 1:1, (b) 2:5, (c) 1:3, (d) 4:1, and (e) 2:9.

Figure 2: XRD spectra of TE products with different Co/Al molar ratios: (a) 1:1, (b) 2:5, (c) 1:3, (d) 4:13, and (e) 2:9.

Figure 3: Expansion behaviors of porous intermetallics with different Co/Al molar ratios: (a) macroscopic, (b) volume, axial, and radial direction expansion.

The various expansion values and different Al contents resulted in the different densities of Co–Al porous intermetallics, as shown in Figure 4. The density of green compacts decreased with increasing Al content

Figure 4: Specimen density before and after the TE reaction with different Co/Al molar ratios.

because the density of Al powder was smaller than that of Co powder. The sample density of each proportion decreased after the TE reaction due to the volume expansion. The density was remarkably changed from 2.77 to 1.86 g cm⁻³ after the TE reaction, which is the minimum density among all specimens because the volume expansion ratio reached the highest value of 76.7% and the highest Al content of 67.3 wt% (Co:Al = 2:9). The density changed from 3.53 to 3.42 g cm⁻³ after the TE reaction, which is the maximum density among all specimens because of the lowest volume expansion of 12.3% and the lowest Al content of 31.4 wt% (Co:Al = 1:1).

3.4 Pore structure analysis

The optical metallographs of Co–Al porous intermetallics are shown in Figure 5. The fusion between Co₂Al₃ particles was poor because of the low T_c (986.4°C) and the short reaction time (about 1 s). Hence, the particles peeled off from the compound surface, and the metallographic structure of this compound could not be observed. The white areas represented the matrix materials of Co–Al intermetallics, while the black areas represented pores. There were big pores among the matrix in all Co–Al porous intermetallics and small pores in CoAl and Co₂Al₉ intermetallics. The big pores (>50 μm) were formed during violent solid–liquid TE reaction. The small pores (<15 μm) could be attributed to the capillary force of liquid Al and finally precipitated from compound particles [5,36]. The values of T_c in specimens with Co:Al = 1:3 and 4:13 were lower (1156.3°C and 1067.1°C) than that in specimens with Co:Al = 1:1 and 2:9 (1327.2°C

Figure 5: Optical microscope images of polished specimens with different Co/Al molar ratios: (a) 1:1, (b) 1:3, (c) 4:13, and (d) 2:9.

and 1420.9°C); hence, the capillary force of liquid Al was small and the small pores in these intermetallics were not obvious. The numbers of pores in Figure 5a and d were relatively more than that in Figure 5b and c. The pores were interconnected and marked with a red line in Figure 5d. Figure 6 shows the microstructure of the fracture surface at different Co/Al molar ratios. Sintered products possessed open-celled characteristics, and these pores are interconnected with each other, especially the big pores among the particles. The pores connectivity was most obvious in Figure 6a and d. Figure 7 shows the open porosity of Co–Al porous intermetallics as a function of Al content. The green compact fabricated by cold extrusion had not to be pressed into a full-density compact. The pores between powder particles Co and Al were important sources of pores after sintering. The open porosities of green compacts were measured to be 17.7, 21.7, 21.2, 18.6, and 16.1% corresponding to Co:Al = 1:1, 2:5, 1:3, 4:13, and 2:9, respectively. The open porosities of Co–Al porous intermetallics increased after sintering, corresponding to the green compacts with the same Co/Al molar ratio. The open porosities were measured to be approximately 43.2, 36.0, 27.1, 32.9, and 42.8% to the specimens with Co:Al = 1:1, 2:5, 1:3, 4:13, and 2:9, respectively. These increased holes formed by the reaction of liquid Al and solid Co during the TE reaction.

EDS was used to confirm the elements in porous intermetallics. Figure 8 shows the elemental compositions of the specimen with Co:Al = 1:1. Figure 8a illustrates the magnification morphology of Co–Al porous intermetallics after grinding and polishing. Figure 8c and d show that Al and Co are distributed evenly on the matrix of the Co–Al compound. Figure 8b shows that porous intermetallic compound mainly included Al and Co element. The EDS point analysis results (as shown in Figure 8e) confirmed that the atomic ratios of Co:Al was 52.14:47.86, which was also close to stoichiometric proportions of nominal compositions. To the ideal result, the atomic ratio of Co:Al should be 1:1 in the sintered specimen, but the EDS result was not consistent completely with the ideal result, which was due to the semi-quantitative analysis of EDS [37,38]. Combined with the XRD pattern (Figure 2a), it was suggested that the matrix should be CoAl intermetallic compound.

3.5 Oxidation properties

Figure 9 shows the mass gain of different Co–Al porous intermetallics changing with time at 650°C in air. All of the mass gain curves exhibited parabolic-like behavior,

Figure 6: SEM fracture micrographs of intermetallics with different Co/Al molar ratios: (a) 1:1, (b) 1:3, (c) 4:13, and (d) 2:9.

indicating that the Co–Al porous intermetallics have superior high-temperature oxidation resistance. The mass gain values after 96 h were 1.57, 6.12, 4.55, 3.13, and 13.50 g m⁻² to the specimens with Co:Al = 1:1, 2:5, 1:3, 4:13, and 2:9, respectively.

Figure 7: Open porosity of Co–Al intermetallics with different Co/Al molar ratios.

The specimen with Co:Al = 2:9 had the largest mass gain during the oxidation process, but it still had the excellent oxidation resistance compared with Ti–Al porous intermetallics (the minimum mass gain was 185.1 g m⁻²) [5]. The specimens with other Co/Al molar ratio showed superior high-temperature oxidation resistance. In particular, the specimens with Co:Al = 1:1 had the least mass gain and the best oxidation resistance. The mass gain came from the oxidation of Co–Al porous intermetallics and unreacted Al and continued to increase because it reacts with oxygen continuously. Although the mass gain had some differences in different Co–Al porous intermetallics, they all showed superior high-temperature oxidation resistance.

4 Conclusion

Co–Al porous intermetallics were fabricated by an efficient and energy-saving method of the TE. There was an obvious heat release phenomenon in the TE reaction. The products of the specimens with Co:Al = 1:1

Figure 8: (a) SEM images, (b–d) elemental mapping and (e) EDS analysis of the porous intermetallic compound with Co:Al = 1:1.

Figure 9: Mass gain of Co–Al porous products oxidized at 650°C in air.

and 2:9 retained their original cylindrical shape without any defects after sintering. The product of the specimen with Co:Al = 1:1 was a single CoAl intermetallic compound, and those with other Co/Al molar ratios were doped with minor or trace ingredients. The intermetallics of Co–Al were mainly CoAl, Co_2Al_5 , $\text{Co}_4\text{Al}_{13}$, and Co_2Al_9 corresponding to their near Co/Al molar ratio. The specimen with Co:Al = 1:1 possessed the highest T_{ig} values of 645.1°C and the highest open porosity of 43.2%. The specimen with Co:Al = 2:9 possessed the highest T_{c} values of 1420.9°C, the lowest density of 1.86 g cm^{-3} , and higher open porosity of 42.8%. All Co–Al porous intermetallics had excellent high-temperature oxidation resistance than other porous intermetallics. We can add some pore-forming agents to increase Co–Al porous intermetallics porosity and decrease its density in future research.

Funding information: This work has been founded by the Fundamental Research Funds for the Central Universities (No. 2019XKQYMS17).

Conflict of interest: Authors state no conflict of interest.

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