

Effect of pore structures on corrosion resistance of porous Ni₃Al intermetallics

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Abstract: Porous Ni₃Al intermetallics were fabricated by elemental powder reactive synthesis method, using carbamide powders as space holders. Corrosion behavior of porous Ni₃Al intermetallics was investigated in a 6 mol/L KOH solution using electrochemical methods and immersion test. Effect of porous structures on the corrosion behavior of the porous Ni₃Al intermetallics was studied. The results indicate that the porous Ni₃Al intermetallics with higher porosities suffer more serious corrosion than the ones with lower porosities because the complicated interconnected porous structures and the large true surface areas exist in the samples with a higher porosity. But the corrosion rates of the porous Ni₃Al intermetallics are not proportional to the true surface areas. The reason is that the pore size, pore size distribution and pore shape of the porous Ni₃Al intermetallics change with the increasing porosity. All the porous Ni₃Al intermetallics with different porosities exhibit excellent corrosion resistance in a strong alkali solution.

Key words: nickel aluminide intermetallic; corrosion; pore structure

1 Introduction

Nickel aluminide intermetallics have been envisaged as good candidates for a number of industrial sectors, including aerospace, automotive, and power generation industries due to their high tensile strength and yield point, low density, and good high-temperature creep resistance [1–4]. Owing to their excellent corrosion and oxidation resistance especially at the high temperature, nickel aluminides are considered as materials that might replace current nickel-based super alloys in rugged environments [5,6].

Recently, porous intermetallics have attracted much attention for their unique properties with the prospect of various potential applications [7–9]. Porous intermetallics such as TiAl, FeAl, and NiAl have been fabricated by sintering elemental powder compacts [10–12]. All of the research works indicate that the oxidation and corrosion resistance of the porous intermetallics are superior to the conventional porous metals [13,14].

However, pores are inherent defects that make the materials mechanically weak while electrochemically

active, there is a decrease in corrosion resistance due to the larger surface area exposed to the electrolyte compared to non-porous materials. But the surface area is not the only factor influencing the corrosion rate of porous structure. The crevices or restrictions of species can result in corrosion rates being not proportional to the true surface area. In a crevice, the solution can become acidic or oxygen deficient, resulting in an autocatalytic process by which localized corrosion accelerates [15]. A previous study [16] has shown that the sintered porous titanium with a higher porosity ratio underwent more severe corrosion than the sample with a lower porosity because of the larger real surface area. However, the unsintered sample with a higher porosity ratio experienced less corrosion than the one with a lower porosity. The discrepancy was related to the pore characteristics of the porous titanium. Furthermore, the electrochemical reaction is essentially a heterogeneous electron transfer reaction occurring at the solid-liquid interface. The three-dimensional natures of the porous Ni₃Al intermetallics significantly increase the complexity of the interfacial structure [17].

Currently, there are only very limited studies on the corrosion characteristics of the porous Ni₃Al

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intermetallics. In a previous study, porous Ni₃Al intermetallics were determined to have better corrosion resistance than the porous Ni and Ti alloys [13] and could replace the cloth filters in the suspended high enriched alkali solution. In addition, porous Ni₃Al intermetallics were also found to have higher electrocatalytic activity during hydrogen generation of alkaline water electrolysis than porous Ni [14]. During filtration and hydrogen revolution reaction process, different pore structures of the materials will be used. So, the study of the effect of the pore structures on corrosion resistance of porous Ni₃Al intermetallics is essential.

The present work aims to clarify the influences of the pore structures on the corrosion behavior of the porous Ni₃Al intermetallics during electrochemical and immersion tests, and this was accomplished through a systematic comparison of the corrosion characteristics of the porous Ni₃Al intermetallics. The porous Ni₃Al intermetallics with porosity ranging from 38% to 60% were prepared and their corrosion characteristics in a 6 mol/L KOH solution were investigated.

2 Experimental

2.1 Preparation and characterization of porous Ni₃Al intermetallics

The porous Ni₃Al intermetallics were fabricated through reactive synthesis of Ni (38 μ m) and Al (38 μ m) elemental powders. In order to get a higher porosity, high-purity sphere carbamide powders (~50 μ m) as the space holders were added. The porosity of Ni₃Al intermetallics was tailored by adjusting the amount of carbamide powders added to the samples. The mixed powders were pressed to compacts with dimensions of 5 mm×45 mm×1 mm under a pressure of 200 MPa. Then the specimens were sintered in a vacuum furnace under 1×10^{-3} Pa at 1250 °C for 2 h followed by furnace cooling to room temperature. There were four types of samples with the porosities of 38%, 50%, 54% and 60% fabricated by adding carbamide powders of 0, 5%, 10% and 15% (mass fraction), respectively.

The morphology and composition as well as crystalline structures of the fabricated porous Ni₃Al electrode were characterized by a field-emission scanning electron microscope (NOVA NANOSEM 230) and X-ray diffractometry (XRD, D/MAX-255). The open porosity was measured by the Archimedes method [10]. The gas permeability and maximum pore size were determined on an FBP-3III porous material test instrument, and 99.9% (volume fraction) purified N₂ was used as the fluid medium [11]. The maximum pore size could then be measured by the bubble point method [12].

2.2 Electrochemical measurements

2.2.1 Sample preparation for corrosion test

Electrical connection to the porous Ni₃Al intermetallics was achieved by attaching a copper wire to one side of the samples at mid-height. The electrical connection was confirmed by means of a multimeter. The specimens were next encapsulated in a thermosetting polyester resin that was allowed to cure for 24 h at room temperature before use. Before the resin was poured over the specimens, a polytetrafluoroethylene tape was used to cover the test face, which could eliminate the process of grinding off the resin after curing. The exposure area was 1 cm². Finally, insulating lacquer was applied at the resin specimen interface to prevent seepage of electrolyte down the sides of the specimen [18].

2.2.2 Electrochemical test procedure

Electrochemical measurements were performed using the standard three-electrode system. The working electrode was porous Ni₃Al intermetallics with both sides exposed to the solution. The reference electrode was Hg/HgO reference electrode and a platinum sheet with 2 cm² surface area was used as a counter electrode. The test electrolyte was 6 mol/L KOH (analytical reagent) in deionized water.

The potentiodynamic polarization tests and electrochemical impedance spectroscopy (EIS) analysis were performed after 24 h immersion at open circuit. Potentiodynamic curves were measured by scanning the potential from -0.4 V below the open circuit potential to +1.5 V at a scan rate of 0.02 V/s. Each electrochemical experiment was repeated three times with a fresh specimen for each test. The corrosion potential and the corrosion current density were obtained through Tafel approximation. The EIS measurements were obtained using a polarization of ± 0.01 V in the frequency range of 10⁵ Hz to 10⁻¹ Hz and 6 points measuring per decade. EIS spectra were interpreted by the software ZSimpWin 3.10.

2.3 Immersion test

The variation of the corrosion rate at 25 °C for porous Ni₃Al with different porosities was evaluated by measuring the relative mass loss vs time in a 6 mol/L KOH solution. The samples were ultrasonically cleaned in distilled water for 20 min and then dried at 90 °C for 20 min before being weighted on an analytical balance with a resolution of 0.0001 g. The corrosion attack was expressed in terms of relatively mass loss.

3 Results

3.1 Characterization of porous Ni₃Al intermetallics

The fabricated porous Ni₃Al was examined by

X-ray diffraction to identify its phase composition and crystalline structure. Figure 1 shows the XRD patterns of powder compacts at different temperatures. As the temperature rises, peaks related to Ni_2Al_3 , NiAl and Ni_3Al phases appear, while the Ni and Al peaks become weakened. The formation of intermediate phase during the sintering procedure results from the interdiffusion reaction between Ni and Al atoms in compacts. When the temperature reaches 1250°C , single Ni_3Al phase appears on the entire compact. Six peaks in the diffraction spectra at 25° , 37.5° , 44° , 51° , 57.5° and 75° can be assigned to (100), (110), (111), (200), (210) and (220) planes of Ni_3Al , respectively.

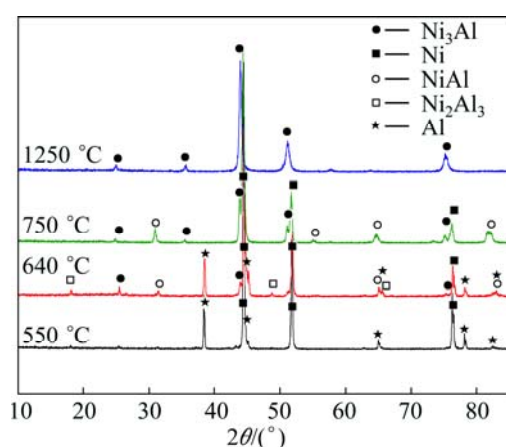


Fig. 1 XRD patterns of powder compacts at different temperatures

The pore structure parameters of porous Ni_3Al are listed in Table 1. As the carbamide content increases, the maximum pore size, permeability and porosity increase respectively. As well known, some small pores exist in the green compact during the pressing procedure and this kind of pores are very small. During the sintering process, carbamide was decomposed below 200°C , and large pores were formed. In addition, Al atoms diffused towards Ni atoms because of the discrepancy diffusion rate between Ni and Al elements [19]. This was the Kirkendall effect [20]. Pores were formed in the original sites of Al atoms. When the temperature increased, the diffusion rate of Al increased to promote the formation of Kirkendall pores, and a small amount of remained Al

Table 1 Pore structure parameters of as-prepared porous Ni_3Al electrode

Mass fraction of carbamide/%	Maximum pore size/ μm	Porosity/%	Permeability/ $(\text{m}^3 \cdot \text{kPa}^{-1} \cdot \text{s}^{-1})$
0	10.2	38	128
5	12.6	50	166
15	65.7	54	512
20	92.0	60	1466

powders turned into liquid phase and thus a lot of middle sized pores were formed. In porous Ni_3Al intermetallics, the amount of small sized and middle sized pores was the same, and the amount of large sized pores increased with increasing the carbamide content.

3.2 Electrochemical measurements

The true surface areas of the porous samples are often determined by cyclic polarization curves [21,22]. Figure 2(a) shows the cyclic voltammogram of porous Ni_3Al (Porosity of 38%) at different scan rates to determine the double layer capacity of the electrodes. From Fig. 2(a), stationary double layer currents can be seen between the potential ranging from 0 to 0.01V (vs SCE). In this region, the average of capacitive current is proportional to the double layer capacitance [21], and the variation of the average of double layer current densities $J_{\text{dl,ave}} = (J_c + J_a)/2$ as a function of potential sweep rate can be described as

$$J_{\text{dl,ave}} = C_{\text{dl}}(d\phi/dt)$$

where C_{dl} is the double layer capacitance of electrode, and J_c and J_a are cathodic and anodic current densities,

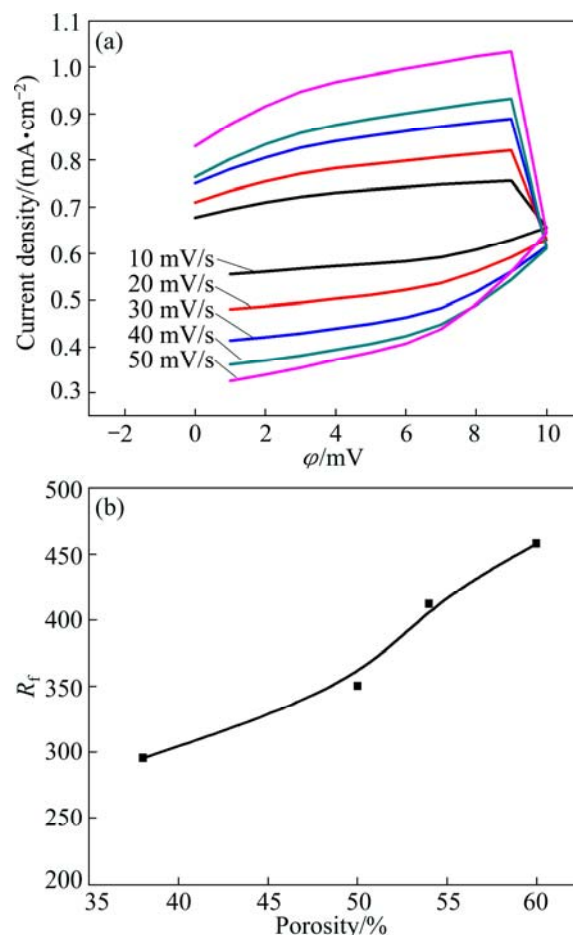


Fig. 2 Cyclic voltammograms in double layer region of porous Ni_3Al intermetallics with porosity of 38% at scan rates ranging from 10 to 50 mV/S (a) and relationship between roughness factor and porosity of porous Ni_3Al intermetallics (b)

respectively. The relative magnitude of the respective roughness factor, R_f , which stands for the real surface of the electrode [22], is calculated by assuming the value of $20 \mu\text{F}/\text{cm}^2$ as the capacitance of smooth mercury electrode and the results are summarized in Fig. 2(b). The results indicate that the roughness factors or real surface areas of all the samples are between the value of 300 and 400, and the order is: $R_f(\text{porosity } 38\%) < R_f(\text{porosity } 50\%) < R_f(\text{porosity } 54\%) < R_f(\text{porosity } 60\%)$, indicating that the porous Ni_3Al intermetallics with a higher porosity have a larger active surface area but the increasing extent is just within certain limits.

Figure 3 presents the evolution of the open circuit potential as a function of time. As it can be seen, the open circuit potentials of the samples experience a sharp rate of change and stabilize in a relatively short period of time. This shift is an indication of the passivity of electrode surface. In this work, higher positive open circuit potential exhibits for the porous Ni_3Al intermetallics with lower porosity. This indicates that the corrosion resistance of the samples with a lower porosity is better.

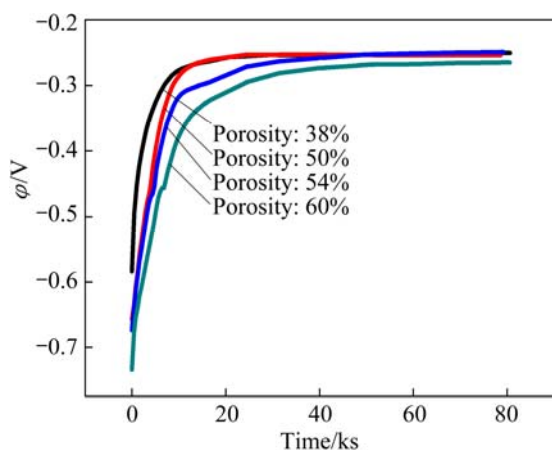


Fig. 3 Evolution of open circuit potential over time for porous Ni_3Al intermetallics in 6 mol/L KOH solution

The corresponding changes of potentiodynamic polarization for the porous Ni_3Al intermetallics with different porosities are shown in Fig. 4, and the extracted electrochemical parameters are listed in Table 2. The J_{corr} values were determined by extrapolating the linear Tafel segments of the anode and cathode polarization curves and $J_{\text{corr}}/S_{\text{real}}$ values [23] reveal the true corrosion current of the porous Ni_3Al intermetallics. It can be seen that porous Ni_3Al with lower porosity has higher free corrosion potential and lower corrosion current than the one with higher porosity. In fact, the value of free corrosion potential represents a thermodynamic characteristic of a given metal-electrolyte system, but not the kinetics of material corrosion. The J_{corr} value can more accurately reflect the corrosion rate than ϕ_{corr} .

Therefore, the increase of J_{corr} values and $J_{\text{corr}}/S_{\text{real}}$ values of porous Ni_3Al intermetallics with higher porosity corresponds to the significantly higher anodic kinetics and lower cathode kinetics. It can speculate based on Ref. [24] that NiO , NiOH and/or NiOOH may exist between the potential ranges of -0.2 V and $+1.0 \text{ V}$, which is supported by the results reported on NiCoZn electrode [25].

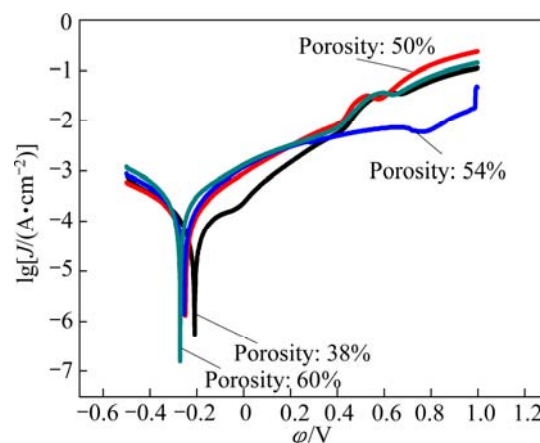


Fig. 4 Polarization curves of porous Ni_3Al intermetallics after immersing in 6 mol/L KOH solution for 24 h

Table 2 Corrosion parameters obtained from potentiodynamic polarization curves

Porosity of $\text{Ni}_3\text{Al}/\%$	$\phi_{\text{corr}}/\text{V}$	$J_{\text{corr}}/(\text{A}\cdot\text{cm}^{-2})$	R_f	$(J_{\text{corr}}\cdot S_{\text{real}}^{-1})/(\text{A}\cdot\text{cm}^{-2})$
38	-0.209	8.124×10^{-5}	295	2.75×10^{-7}
50	-0.249	1.033×10^{-4}	350	2.95×10^{-7}
54	-0.254	1.288×10^{-4}	412	3.12×10^{-7}
60	-0.271	2.056×10^{-4}	458	4.48×10^{-7}

The typical surface morphologies of the porous Ni_3Al intermetallics samples polarized to a potential of 1.0 V from -0.5 V are shown in Fig. 5. As compared with the material before electrochemical tests, the changes of the pore structure are not obvious and there is also no pit corrosion or loosen corrosion product on the surface of the porous Ni_3Al intermetallics, which exhibits excellent corrosion resistance of all porous Ni_3Al intermetallics with different porosities in KOH solutions.

Impedance spectra of porous Ni_3Al intermetallics with different porosities after immersion in a 6 mol/L KOH solution for 24 h are presented in Fig. 6, where all the samples exhibit similar spectral features. Capacitive behavior represented by the phase angle approaching -90° and typical of in passive materials, appears in a medium to low frequency range, which means that a passive film forms on all samples in the electrolyte.

The large phase angle peak can be indicative of the

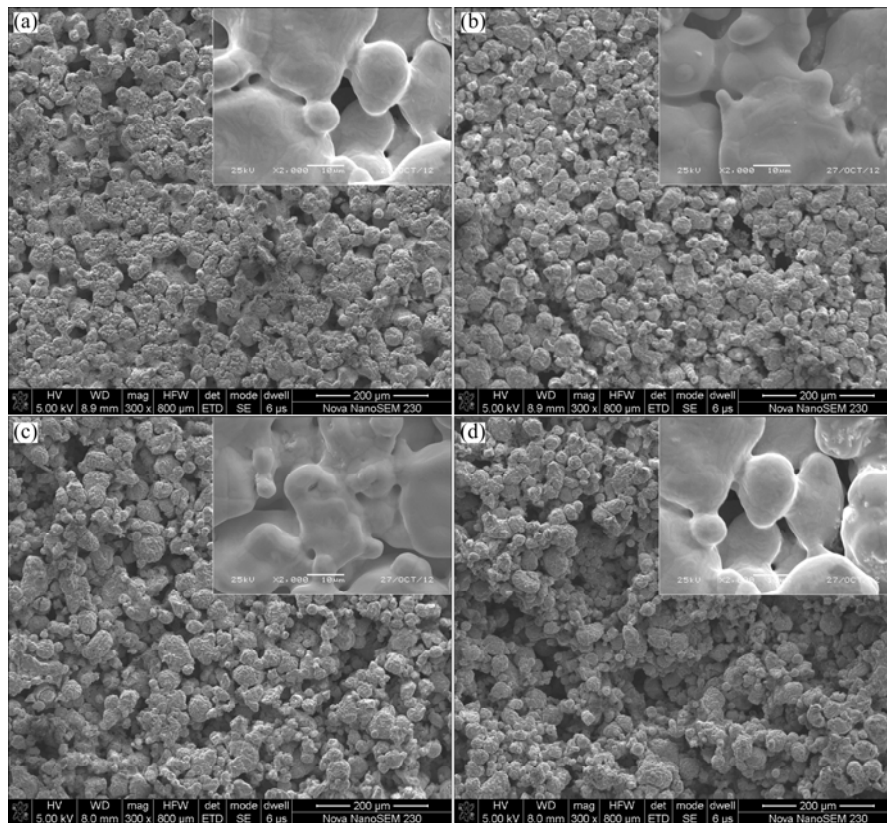


Fig. 5 Typical SEM surface morphologies of porous Ni_3Al intermetallics with different porosities before (a, c) and after (b, d) potentiodynamic polarization measurement in 6 mol/L KOH solution, terminated at 1.0 V: (a, b) 38%; (c, d) 60% (Modified morphologies of sample are shown in insets)

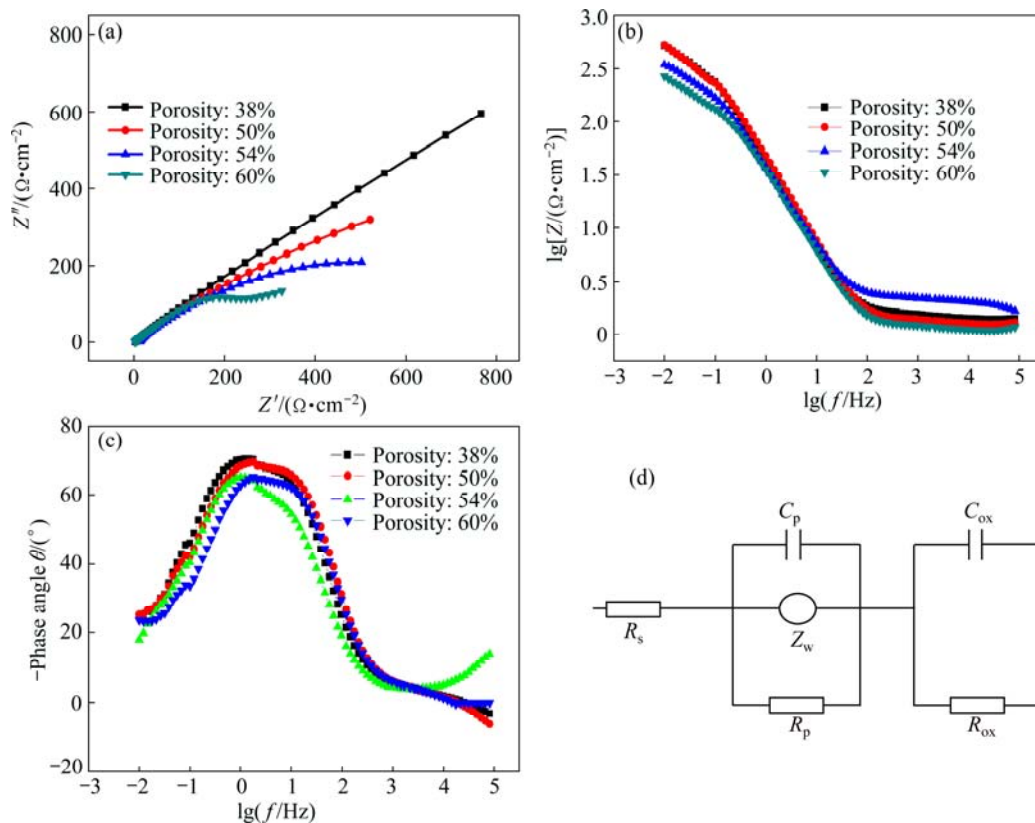


Fig. 6 Nyquist plots and Bode plots of porous Ni_3Al intermetallics with different porosities in 6 mol/L KOH solution: (a) Nyquist plots; (b) $\lg Z$ vs $\lg f$; (c) Phase angle vs $\lg f$; (d) Equivalent circuit

interaction of at least two time constants. Therefore, an equivalent circuit is proposed to model the EIS data obtained from the samples, as shown in Fig. 6(d). In this model, R_s is the resistance of the solution, R_p is the additional resistance of the solution inside the pores, R_{ox} is the charge transfer resistance of the oxygen layer, C_p is the capacitance of the pore wall and C_{ox} is the capacitance of the oxygen layer [25]. It should be mentioned that both capacitances used in the equivalent circuit are not ideal and the deviation from the ideal behavior has been taken into account using a constant phase element CPE [$Z_{CPE}=A_{CPE}(j\omega)^{-n}$]. The appearance of the CPE element is often related to the electrode roughness or to the inhomogeneity in the conductance or dielectric constant [26,27]. However, the data cannot be explained with an only two $R-C$ circuit and the equivalent circuit has to be modified by diffusion impedance parallel to the R_p-C_p circuit. For porous materials, corrosion product film forms not only on the metal surface but also on the internal surroundings of pores. So, a diffusion process exists.

The electrical parameters of the equivalent circuit are listed in Table 3. It can be seen that all the R values decrease with the increase of the porosity of porous

Ni_3Al intermetallics, which shows that the porous samples with lower porosity have better corrosion resistance. Furthermore, a lower capacity is visible in samples with lower porosity. The Warburg parameters (W) are all very small, which means that in these samples, diffusion process was weak and not the nominate step. Since the majority of pores of the porous Ni_3Al intermetallics are interconnect (Fig. 7), interconnected channels allow the free flow of the liquid into the pores.

3.3 Immersion test

The mass changes after immersion test in a 6 mol/L KOH solution for 480 h are shown in Fig. 8 and the changes of pore characterization are listed in Table 4. They both exhibit that the porous Ni_3Al intermetallics with higher porosity suffer more corrosion than the one with lower porosity. After 480 h corrosion process, the mass change of the materials with the porosity of 60% and 38% are 1% and 0.05%, respectively, which is far smaller than 2%. And the pores characteristics of the materials almost remain the same. It shows that porous Ni_3Al intermetallics exhibit excellent corrosion resistance.

Table 3 Fitting results of EIS measurements

Porosity of Ni_3Al /%	$R_s/(k\Omega \cdot cm^2)$	$R_p/(k\Omega \cdot cm^2)$	$C_p/(\mu F \cdot cm^{-2})$	$W/(k\Omega \cdot s^{-1})$	$R_{ox}/k\Omega \cdot cm^2$	$C_{ox}/(\mu F \cdot cm^{-2})$
38	0.439	819.3	6	9.59	50.98	8
50	0.465	607.6	9	1.304	42.86	11
54	0.480	520.6	11	1.46	40.46	12
60	0.540	412.3	12	1.353	38.16	16

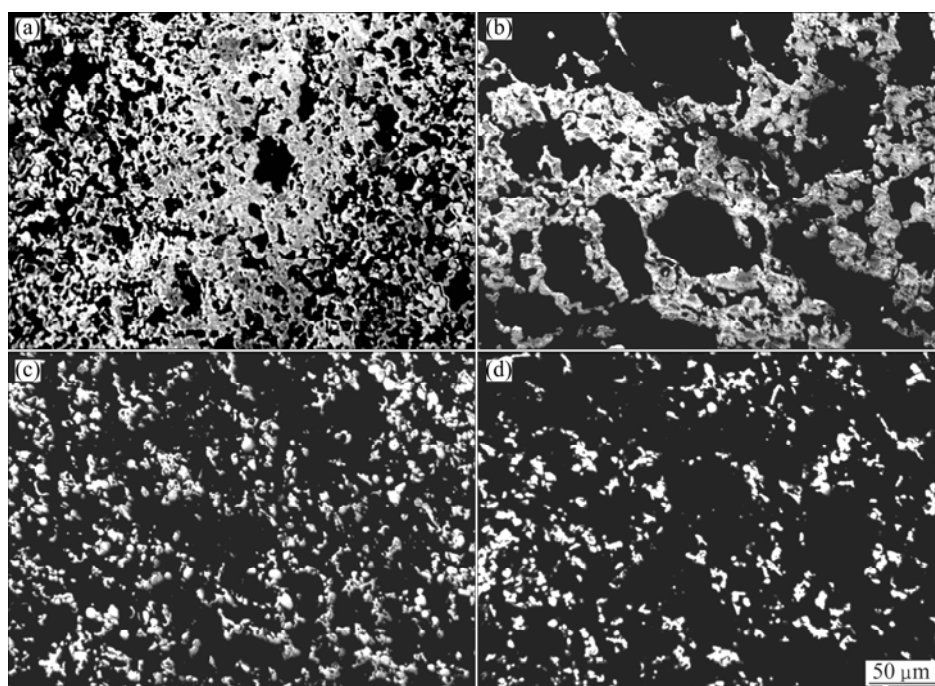


Fig. 7 Optical micrographs of porous Ni_3Al intermetallics with different porosities: (a) 38%; (b) 50%; (c) 54%; (d) 60%

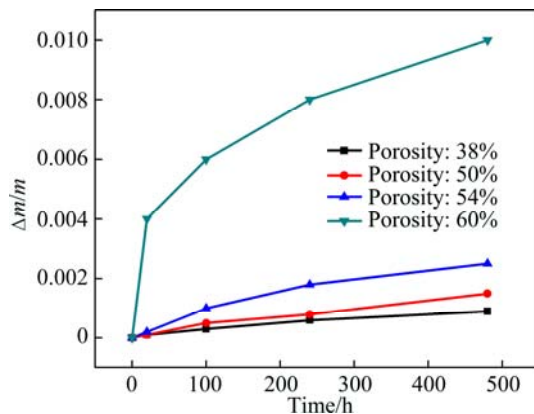


Fig. 8 Corrosion kinetic curves of porous Ni₃Al intermetallics in 6 mol/L KOH

Table 4 Pore structures of porous Ni₃Al intermetallics after immersion test

Mass fraction of carbamide/%	Maximum pore size/ μm	Porosity/%	Permeability/ $(\text{m}^3 \cdot \text{kPa} \cdot \text{h}^{-1} \cdot \text{m}^{-2})$
0	10.2	38.5	133
5	12.7	51.5	175
15	69.5	55.8	532
20	97	62.6	1496

4 Discussion

From the electrochemical results and immersion test, porous Ni₃Al intermetallics with higher porosity and larger pore size have poorer corrosion resistance. This is to a certain extent expected from the larger real surface area. The changes in electrochemical parameters obtained from the polarization curves of the porous Ni₃Al intermetallics with increasing porosity and pore size are summarized in Table 1. It shows that the corrosion rates are not proportional to the true surface area. In fact, with the increase of the porosity, the surface area of the porous Ni₃Al intermetallics increases, the pore size increases and the surface quality and structure uniformity change. In a small pore, the solution can become acidic or oxygen deficient, resulting in an autocatalytic process by which localized corrosion accelerates. In addition, it is known that the corrosion resistance of the Ni₃Al intermetallics is associated with the formation of the nickel oxide films on the alloy's surface. The surface quality, the amount of the residues left on the surface and the microstructure inhomogeneity of the porous Ni₃Al intermetallics are the critical factors affecting the formation and quality of the oxide films. However, the preparation of the porous Ni₃Al intermetallics generally consists of complex thermomechanical processes. From the optical and SEM micrographs of porous Ni₃Al intermetallics, it can be seen that with the increase of the porosity, many pores exhibit irregular shape. And the inner walls of the pores

are generally unsmooth. These features may also be vulnerable sites and imperfections in any passive films [20].

There are three types of pores in the porous Ni₃Al intermetallics. The large pores with sizes of 100–200 μm are primarily formed by the decomposition of the space-holder, carbamide particles. The middle pores with sizes of 10–20 μm can be attributed to the Kirkendall diffusion between Ni and Al atoms. The small pores with pore sizes below 10 μm are formed by the trapped residuals of carbamide decomposition and exist in the green-compact [11]. The small pores show a better size-uniformity and a more uniform distribution than the large ones. When the porosity increases, the interconnectivity of larger pores is clearly increased.

For the samples with a lower porosity, the majority of the pores are the middle pores and small pores, which are unlikely to have trapped appreciable volumes of solution. This causes the samples relative resistance to induce corrosion. When the porosity increases, the interconnectivity of the larger pores is markedly improved. The interconnected channels allow the free flow of the liquid and fewer sites are available to induce corrosion. Furthermore, with the change of the porosity, pore size distribution and pore shape of the porous Ni₃Al intermetallics change correspondingly. It is difficult to provide a complete characterization of the pores by solely measuring the sample porosity and real surface area. This also accounts for that the corrosion rates are not proportional to the true surface area.

5 Conclusions

- 1) Porous Ni₃Al intermetallics with different porosities exhibit excellent corrosion resistance in strong alkali solution.
- 2) The samples with higher porosities suffer severer corrosion than the ones with lower porosities because the real surface areas of porous Ni₃Al intermetallics generally increase with the increasing porosities.
- 3) The corrosion rates are not proportional to the true surface areas of porous Ni₃Al intermetallics because the surface area is not the only factor influencing the corrosion rate. The pore size, pore size distribution and pore shape should also be taken into account.

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孔结构对 Ni_3Al 金属间化合物多孔材料抗腐蚀性能的影响

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摘 要: 采用元素粉末反应合成的方法并用尿素作为造孔剂制备 Ni_3Al 金属间化合物多孔材料。用电化学和浸泡实验表征 Ni_3Al 金属间化合物多孔材料在 6 mol/L KOH 溶液中的抗腐蚀行为。系统研究孔结构对材料抗腐蚀性能的影响。研究表明: 孔隙率较大的 Ni_3Al 金属间化合物多孔材料较孔隙率较小的样品腐蚀更严重, 这是由于孔隙率较大的样品具有复杂的联通孔结构以及较大的比表面积。然而, 材料的腐蚀速率与比表面积并不成正比, 这是因为随着孔隙率的增大, 材料的孔径大小、孔径分布以及孔隙形状都随之变化。不同孔隙率大小的 Ni_3Al 金属间化合物多孔材料在碱溶液中均表现出较好的抗腐蚀性能。

关键词: 镍铝金属间化合物; 腐蚀; 孔结构

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