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Application of porous TiAl intermetallic compound in purification of industrial TiCl₄ liquid in Ti metallurgy

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Abstract

Industrial TiCl₄ liquid, which is a key intermediate in Ti metallurgy, is hard to be purified by traditional porous metals or ceramics due to its characteristics of easy hydrolysis, toxicity and corrosivity. In this work, porous TiAl intermetallic filter tubes were fabricated through a reactive synthesis process, and their filtration properties in industrial TiCl₄ liquid were investigated. The cyclic corrosion kinetics of porous TiAl in TiCl₄ follows a parabolic rate law, in which the corrosion rate constant is 4.8%, 0.06% and 0.04% of that of porous Ti, Ni and 316L stainless steel, respectively. After the cyclic corrosion of 408 h, the maximum aperture reduction rate of porous TiAl is 3.9%, which is far lower than that of porous Ti, Ni and 316L, indicating good pore structure stability. Porous TiAl filter tubes exhibit clean filtration performance in the solid-liquid separation of raw TiCl₄ suspension with stable filtrate solid content of 0.1 wt% and high filtration flux of 8.17 m³·m⁻²·h⁻¹ at the maximum aperture of 17.0 μm. The filtration equation and the filter medium resistance of porous TiAl are determined.

1. Introduction

Porous intermetallic compounds have been studied in depth due to their property advantages of both metal and ceramic materials [1–3], such as easy machining and assembly properties [4], good thermal shock resistance [5] and corrosion resistance to rugged environments [6–8], etc. The special properties of the new porous material mainly come from its highly ordered structure and the mixed bonding characteristic of the combination of metallic and covalent bonds [3]. Significantly, porous intermetallic compounds fabricated through a reactive synthesis process have good open pore structure [9–11], which can be applied in filtration and purification procedure [1, 6, 12, 13], including solid-liquid separation in highly corrosive environments [12] and solid-gas separation in high temperature fields [6]. In the application fields of harsh environments, traditional porous metals exhibit insufficient corrosion resistance, and porous ceramics show poor thermal shock resistivity and machinability, which limits their applications.

At present, porous intermetallic compounds mainly consist of three types: (1) Ti based intermetallics, including TiAl [2, 14, 15], Ti₃Si [16, 17], Ti₃SiC₂ [13] and Ti₃AlC₂ [18], (2) Fe based intermetallics, including FeAl [19] and FeAlCr [6] and (3) Ni based intermetallics, including Ni₅Al [8] and Ni₅Al-Mo [20]. Among them, porous Ti based intermetallics have good corrosion resistance to strong acid solution [1], porous Fe based intermetallics have good oxidation and sulfidation resistance at high temperature [6], and porous Ni based intermetallics have good electrochemical catalytic performance [7]. These unique properties indicate their potential applications. As a typical example of porous Ti based intermetallics, porous TiAl and its membrane prepared by reactive synthesis exhibit good pore structure controllability [9, 21, 22], excellent corrosion resistance to strong acid and high temperature oxidation resistivity [1], which gives this material a good application prospect in the separation and purification field of highly corrosive or toxic substances, such as industrial TiCl₄ suspension [1].

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Solid-liquid separation of TiCl₄ suspension is a key step in the process of magnesium reduction of TiCl₄ to produce Ti metal [23–25]. TiCl₄ is a colorless or slightly yellow liquid with strong pungent acid odor at room temperature, and is easily hydrolyzed with water to generate HCl and smoke in the air, which belongs to highly toxic and corrosive chemicals [26]. TiCl₄ shows subsequent strong corrosivity after hydrolysis, which requires the filter material to have excellent corrosion resistance and good machinability and sealability. However, traditional porous metals or ceramics are difficult to be used in the separation process due to the lack of comprehensive performance. Filter cloth was widely used in this process [1], which shows low filtration accuracy and poor anti-TiCl₄ corrosion stability, resulting in frequent replacement of filter elements, hydrolysis of a large amount of TiCl₄, and serious environmental pollution [26]. Porous TiAl intermetallic compound can be used for TiCl₄ solid-liquid separation, which can solve the matching problem between filter material properties and harsh environment [1]. To the best of our knowledge, further investigations on the corrosion resistivity to industrial TiCl₄ liquid and filtration application for raw TiCl₄ suspension of porous TiAl intermetallic compound have not been observed in the literature.

In this work, porous TiAl intermetallic compound was prepared by cold isostatic pressing and reactive synthesis of elemental powders. The porous material was used as the filter element for the end filtration process of raw TiCl₄ suspension with a solid-liquid ratio of ca. 8 wt% combined with filter-backwash technology in a Ti metallurgy factory. The corrosion resistance and filtration procedure of porous TiAl were discussed in details. The filtration equation for this new porous material in solid-liquid separation process was established.

2. Experimental procedure

Ti and Al elemental powders with the median diameter of 30–35 μm and the purity over 98.5% were used as raw materials. Dry mixing of the powders was carried out in a drum-type mixer with the time of 8 h. The nominal composition is Ti-35 wt%Al, which is within the single-phase region of the γ-TiAl phase [9]. Flaky and tubular green compacts were formed by die pressing and cold isostatic pressing, in which tool steel and synthetic rubber were used as the die materials, respectively. The forming pressure is 200 MPa and the holding time is 10 s. No lubricant was applied on the die surface due to the good compactibility of Ti and Al elemental powders. Vacuum sintering with Mo metal as the heating element was used for the reactive synthesis procedure with the pressure in the range of $1 \times 10^{-2}$–$1 \times 10^{-4}$ Pa. A stepwise heating process [27] was utilized in the synthesis procedure with the final sintering temperature of 1300 °C for 30 min followed by a furnace cooling process. The maximum pore size of the synthesized porous TiAl was determined by the bubble-point method [28]. The open porosity was measured by the Archimedes method [9]. The microscopic pore structure was observed by scanning electron microscopy (SEM, JEOL JSM-5600LV).

Flaky sintered compacts of porous TiAl were utilized to test their corrosion behavior in industrial TiCl₄ liquid at room temperature through a cyclic corrosion method [8, 29]. After each corrosion cycle, the test samples were immediately and repeatedly rinsed with plenty of water, followed by an ultrasonic cleaning for 30 min to remove the corrosion resultants and hydrolysis products. The corrosion weight loss per unit area of each sample was measured by the weighing method to determine the corrosion kinetics curve. The maximum pore size of each sample was measured by the bubble-point method to ascertain the pore structure stability with the corrosion time. Traditional porous metals, including Ti, Ni and 316L stainless steel, which are widely used at present, were used for the comparison of the corrosion resistance against TiCl₄ liquid.

Porous TiAl filter tubes were used to conduct the solid-liquid separation process of raw TiCl₄ suspension in the purification workshop. The raw TiCl₄ was obtained by the reaction of enriched titanium ore and petroleum coke in fluidized chlorine gas flow at 900–1000 °C. The cooled raw TiCl₄ is a reddish-brown suspension with the solid content of 8 wt%–10 wt%. The main compositions of this suspension are shown in Table 1. The main impurities are Cl₂ and high valence state ion chlorides, which are highly corrosive. The end filtration method [30–32] combined with the filter-backwash technology was adopted in the filtration procedure. The backwash cycle is set to 1 h and the washing time is 1 min. During the continuous filtration with the total duration of 30 days, the filtration flux of the porous TiAl filter tubes and the solid content of the filtrate were measured.

| Table 1. Composition of raw TiCl₄ suspension liquid. |
| --- | --- | --- | --- |
| Composition | Cl₂ | SiCl₄, AlCl₃, FeCl₃, VOCl₃, TiOCl₂, etc | Suspended solids | TiCl₄ |
| Content/% | 0.05–0.3 | 0.08–0.65 | 8–10 | balance |
3. Results

Porous intermetallic compound with nearly single γ-TiAl phase (a = 4.005 Å and c = 4.070 Å, Space Group P4/mmm, PDF 65-8566) can be obtained through the reactive synthesis process of Ti and Al elemental powders, as shown in figure 1(a) [2]. In addition, due to the sintering loss of Al element, trace Ti₅Al phase [2] (a = 5.793 Å and c = 4.649 Å, Space Group P6₃/mmc, PDF 52-0859) exists in the sintered compact. The characteristic of high-purity intermetallic compound is beneficial to the corrosion resistance of the material. Figures 1(b)–(d) show the porous TiAl filter tube and its microscopic pore structure, respectively. The prepared porous TiAl tube has the dimension of Ø50 mm × 330 mm (figure 1(b)). The open porosity of the porous material is measured to be 45%. The maximum pore size is determined to be 17.0 μm, which is consistent with the results in figure 1(c). A large number of connected pores are generated amongst the resultant particles with the size of pores generally in the range of several tens of micrometers. Meanwhile, there are a few subround pores in section with several micrometers inside the resultant particles. The generation of such connected coarse pores and internal fine pores is mainly due to the Kirkendall effect at different stages of element diffusion and reaction [1, 9]. The pores in the synthesized porous TiAl compound show smooth surface characteristics (figure 1(d)), which indicates that sufficient particle surface diffusion behavior exists during the sintering procedure. In fact, the reactive synthesis process of elemental powders has the strong surface diffusion behavior, which is related to the high diffusion rate of elemental metals [1, 3] and the large concentration gradient in the mixed powders. Obviously, the smooth porous surface facilitates the permeability of the porous material.

Figure 2 shows the corrosion kinetics curves of porous TiAl and traditional porous materials including Ti, Ni and 316L stainless steel in TiCl₄ liquid with the total corrosion time of 408 h. The corrosion procedure of porous TiAl in TiCl₄ is similar to that in strong acid [29], in which the loss in weight per unit area of porous TiAl is stable and almost remains constant in the later stage of corrosion. However, the weight loss of porous Ni and 316L stainless steel has a large change range, and their variation trends show the characteristic of instability. The kinetics curves of the porous materials are fitted according to the linear and parabolic rate laws, respectively, to determine the corrosion characteristics and the corresponding corrosion rate constants (table 2). The fitting degree of the parabolic law is obviously higher than that of linear law for the corrosion process of the porous materials with their parabolic determination coefficients all close to 1. The corrosion rate constant of porous TiAl is 4.8%, 0.06% and 0.04% of that of porous Ti, Ni and 316L stainless steel, respectively, showing good corrosion resistance to TiCl₄. After the entire cyclic corrosion, the weight loss of porous TiAl is only 0.02 g · m⁻², which is 2.2% and 2.6% of that of porous stainless steel and Ni, respectively. Porous Ti metal, on the other hand, shows a corrosion weight gain behavior with the mass gain of 0.08 g · m⁻² in total. This is mainly caused by the adhesion between Ti metal and solid resultants, such as corrosion or hydrolysis products, which are difficult to remove. The weight gain behavior is not conducive to the recoveries of pore size and flux for the porous material, which is further confirmed in subsequent tests of pore structure stability.

Figure 3 shows the change behavior of the maximum pore size of porous TiAl with corrosion time compared with porous Ti, Ni and 316L stainless steel. The slight corrosion weight loss of porous TiAl ensures the stability of its pore structure to a certain extent. In the procedure of cyclic corrosion, the variation of pore size can be used to characterize the effectiveness and stability of pore structure in the filtration process. The maximum pore size of porous TiAl remains almost unchanged at 17 μm, however, the ones of porous Ti, Ni and 316L stainless steel decrease linearly with the corrosion time. After the cyclic corrosion of 408 h, the maximum pore size of porous TiAl decreases by 3.9%, which has little influence on the filtration efficiency. However, the maximum apertures of porous Ti, Ni and 316L stainless steel decrease by 69.6%, 92.3% and 94.1%, respectively. The pore structures of porous Ni and stainless steel have basically failed. The pore size of porous Ti decreases rapidly with the corrosion time, and the decrease rate is the largest among the four, which is mainly related to the corrosion weight gain behavior mentioned above (figure 2). It can be seen from the pore size change behavior that the variation of the maximum pore size of porous Ti is still in an unstable state at the later stage of the corrosion process, showing poor anti-TiCl₄ corrosion performance. During the cyclic corrosion procedure, the weight loss and pore size of porous TiAl remain almost unchanged, while porous Ti, Ni and 316L stainless steel show rapid or even complete failure of the pore structure, which ensures the application of porous TiAl in harsh environments.

Figure 4 shows the variation of TiCl₄ filtration flux with time for porous TiAl filter tubes during a 30-day continuous filtration process. The filtration flux of porous TiAl tubes fluctuates slightly around 8.17 m³ · m⁻² · h⁻¹ during the filtration cycle, which indicates a characteristic of stable and high flux. The solid content of the filtrate is around 0.1 wt%, which is far beyond the requirement of 0.5 wt% for the production factory. The comparison of raw TiCl₄ suspension, the filtrate through filter cloth bags and that through porous TiAl filter tubes is shown in figure 5. The raw TiCl₄ presents a dark brown suspension with the solid content of ca. 8 wt%. The filtrate purified by porous TiAl presented a primrose yellow and transparent state with the solid content of 0.1 wt%. The filtrate filtered by the cloth bags is still in suspension state with the solid content of about
Figure 1. XRD pattern of TiAl sintered compact (a) [2], image of the synthesized TiAl filtration element (b) and pore microstructure of porous TiAl intermetallic compound (c), (d).
0.5 wt%. Porous TiAl filter tubes show both high filtration accuracy and high permeability flux, which helps to achieve a long-term and stable filtration process. The cloth bags currently used in the workshop have the service life of 6–8 days, which results in frequent replacements and causes the massive hydrolysis of TiCl₄. The application of porous TiAl avoided the loss of TiCl₄ filtrate and severe environmental disruption, in which a clean production of Ti metal can be realized.

**Figure 2.** Kinetics curves of porous TiAl during cyclic corrosion in TiCl₄ compared with porous Ti, Ni and 316L stainless steel.

**Table 2.** Comparison of corrosion rate constants and the corresponding determination coefficients R² for porous TiAl, Ti, Ni and 316L stainless steel in TiCl₄.

| Porous material | Linear corrosion rate constant / g·m⁻²·h⁻¹ | R² for linear corrosion | Parabolic corrosion rate constant / g²·m⁻⁴·h⁻¹ | R² for parabolic corrosion |
|-----------------|--------------------------------------------|------------------------|---------------------------------------------|----------------------------|
| TiAl            | 2.73 × 10⁻⁵                                | 0.8357                 | 8.06 × 10⁻⁷                                | 0.9264                     |
| Ti              | 1.85 × 10⁻⁴                                | 0.7902                 | 1.69 × 10⁻⁵                                | 0.9159                     |
| Ni              | 1.56 × 10⁻³                                | 0.9584                 | 1.42 × 10⁻¹                                | 0.9680                     |
| 316L            | 1.87 × 10⁻³                                | 0.8651                 | 2.03 × 10⁻¹                                | 0.9689                     |

**Figure 3.** The maximum aperture variation of porous TiAl during cyclic corrosion in TiCl₄ compared with porous Ti, Ni and 316L stainless steel.
4. Discussion

During the solid-liquid separation procedure by porous filter media, a filter cake layer with a certain thickness exists on the surface of porous media, and the filtration resistance generated by the filter cake layer and the filter medium is the main factors controlling the actual filtration rate \[32\]. Considering the existence of a certain form of filter cake layer in almost all the end filtration processes, there is a four-layer structure, namely, raw liquid/filter cake layer/filter medium/filtrate, in the dynamic model of the filtration procedure of raw TiCl\(_4\) liquid. The Ruth equation \[33–36\] is applied to the filtration procedure, and the relationship of filtration rate with filter media resistance, filter cake layer resistance, filtration pressure difference and TiCl\(_4\) filtrate viscosity is obtained \[36\]:

\[
\frac{d
u}{dt} = \frac{p(1 - s_0)(1 - m\nu)}{\mu\alpha s(\nu + \nu_{in})}
\]

Where, \(\nu\) is the amount of TiCl\(_4\) filtrate per unit area, m\(^3\) · m\(^{-2}\); \(p\) is the filtration pressure difference, Pa; \(\mu\) is the viscosity of TiCl\(_4\) filtrate, Pa · s; \(\rho\) is the density of TiCl\(_4\) filtrate, kg · m\(^{-3}\); \(s\) is the mass concentration of solid particles in raw TiCl\(_4\) suspension; \(s_0\) is the mass concentration of solid particles in TiCl\(_4\) filtrate; \(m\) is the ratio of the total mass to the solid component mass in the filter cake layer; \(\alpha\) is the average filtration specific resistance of

![Figure 4. Permeation flux variation of TiCl\(_4\) suspension liquid through porous TiAl tubes in the filtration plant.](image)

![Figure 5. Filtrate comparison by porous TiAl filter elements and cloth bags.](image)
filter cake layer, m·kg⁻¹; \( \nu_m \) is the filtrate equivalent in the presence of the assumed equivalent filter cake layer, m³·m⁻².

The integral form of the above formula can be further obtained \[^36\] :

\[
(\nu + \nu_m)^2 = \frac{2p(1 - s_o)(1 - ms)}{\mu \alpha \rho s}(t + t_m)
\]

Where, \( t_m \) is the integral constant.

Considering the boundary condition (\( t = 0, \nu = 0 \)) of the filtration process, \( \nu_m \) can be calculated by the relation of \( \nu_m = 2p(1 - s_o)(1 - ms)t_m/ (\mu \alpha \rho s) \). According to the actual measured data of TiCl₄ filtrate amount \( \nu \) with the filtration time \( t \) within 1 h, the corresponding difference value \( \Delta t/\Delta \nu \) is calculated, as shown in table 3. The relation curve between the data of \( \Delta t/\Delta \nu \) and \( \nu \) is shown in figure 6. The corresponding linear fitting result is as follows: \( y = 4.02(x + 104.72) \), \( R^2 = 0.9061 \). The determination coefficient is close to 1, which verifies the applicability of equation (1) in this filtration process. According to equation (2) and the results of figure 6, the average filtration specific resistance of filter cake layer \( \alpha \) is calculated to be \( 3.20 \times 10^6 \) m·kg⁻¹.

The filtrate equivalent \( \nu_m \) is determined to be \( 1.05 \times 10^2 \) m³·m⁻² according to the intercept on the \( \nu \) axis of the linear equation. Thus, the filter medium resistance \( R_m \) of porous TiAl intermetallic compound can be calculated to be \( 6.11 \times 10^{10} \) m⁻¹ according to equation (3) \[^{33, 34, 36}\], which indicates that the porous material exhibits high efficiency in the filtration process.

\[
R_m = \frac{\alpha \rho \nu_m s}{(1 - s_o)(1 - ms)}
\]

The integral constant \( t_m \) can be calculated to be \( 2.20 \times 10^4 \) s according to equation (2) and the value of \( \nu_m \). Therefore, the integral filtration equation for porous TiAl intermetallic compound filter tube in raw TiCl₄ can be determined as follows:

\[
(\nu + 1.05 \times 10^2)^2 = 0.50(t + 2.20 \times 10^4)
\]

5. Conclusion

The reactively synthesized porous TiAl intermetallic compound has the characteristic of nearly single \( \gamma \)-TiAl phase with the open porosity and the maximum aperture of 45% and 17.0 μm, respectively. The cyclic corrosion kinetics of porous TiAl in industrial TiCl₄ liquid follows the parabolic rate law with the smallest corrosion rate
constant compared with that of porous Ti, Ni and 316L stainless steel. In the filtration process of TiCl$_4$ suspension, the TiAl filter element is characterized by high accuracy and performance stability. The filtration equation for porous TiAl in the solid-liquid separation process of raw TiCl$_4$ is determined to be $(\nu + 1.05 \times 10^5)^2 = 0.50(1 + 2.20 \times 10^5)$, and the corresponding filter medium resistance is $6.11 \times 10^{10}$ m$^{-1}$. A long-term filtration process of raw TiCl$_4$ has been realized with porous TiAl tubes as the filter elements, in which severe environmental pollution has been avoided.

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