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Pumping characteristics of Ti-based non-evaporable getter

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Abstract: The application of non-evaporable getters is increasing, they have been widely used in sealed-off vacuum or controlled atmosphere devices. A new type of Ti-based sintered non-evaporable getter has been studied. The room temperature pumping speeds under three activation processes for H_2 were measured as a function of sorbed gas quantities in this paper. At the same time, the optimal activation processes were discussed. The results indicate that the getter combines high porosity and large specific surface area which confirm good performances at room temperature. The threshold of activation temperature is about 500℃ and optimal pumping speed and pumping capacity can be achieved with activation temperature around 600℃ for 30 min. Besides, different configurations can be available in accordance with requirements.

Key words: Ti-based getter; activation; pumping characteristics

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1 Introduction

With the development of science and technology, the getters play more and more important role in industrial and laboratory field. The object of the getters in any vacuum device is permanently to remove any gas released from the constituents of the device during Service life. By so doing the getter maintains the necessary low pressure or gas environment essential for good functioning of the device.

The getters can be divided into two types, namely evaporable getters and non-evaporable getters[1]. Evaporable getters have good room temperature activity, however, adequate volume and a suitable surface on which to deposit the film are needed, the high vapour pressure of barium which through sublimation and migration may cause inter-electrode leakages or stray capacitance effects, etc[2]. As a result, non-evaporable getters (NEGs) are desirable usually.

Non-evaporable getters have been widely used for many years in sealed-off vacuum or controlled atmosphere devices. In most cases, the getters should work at room temperature, and the activation temperature should be as low as possible to avoid thermal damage to surrounding components. Moreover, the main residual gases are water vapor and hydrogen in high vacuum environments[3], the water molecules can be easily removed with bake-out process, but hydrogen cannot be easily pumped dynamically due to its small mass. As a result, hydrogen is the main obstacle to achieve high vacuum. It is necessary to synthesize get-

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ters with high pumping speed and large sorbed quantity for H_2.

2 Experimental

Getters are normally based on alloys from metals of the IVB group and thorium or even on the elements themselves[2]. Ti and Zr are the most common used metal elements for getters. Nevertheless Zr powders have high activity, they are easily flammable and explosive[4]. So Ti powder was chosen as the active element and Mo powder was chosen as the anti-sintering element. Both of them were sintered in high vacuum under high temperature. The Mo powder allows better control of the process, preventing excessive loss of porosity and specific surface area of the material during the sintering[5].

The standard dynamic technique for hydrogen had been adopted for gettering properties tests[2]. The pumping speed (S) and sorbed quantity (Q) could be calculated by formulae as follows:

$$S = F\frac{Pm - Pg}{Pg \cdot A} \qquad Q = \frac{F}{A}\int_0^t (Pm - Pg)\,dt \tag{1}$$

Where S is the pumping speed ($cm^3/s \cdot cm^2$), Q is sorbed quantity during time t ($cm^3 \cdot Pa/cm^2$), Pm is the pressure of the gas injecting chamber (Pa), Pg is the pressure over the getter (Pa), F is the gas conductance (cm^3/s), t is the time for gettering (s), A is the surface area of gettering (cm^2).

Ring samples with 0.62 cm^2 gettering area were adopted in the sorption tests. The sample chamber was pumped until the pressure dropped to 10^{-4} Pa or even lower after loading the samples. The whole system was bakeout to (300 ± 3)℃ to degas. Activation treatment was done after the pressure reached to 10^{-6} Pa. After activation, the getters were allowed to cool down to room temperature before starting sorption tests. Tests were performed at a constant pressure over the getter Pg.

3 Results and discussions

3.1 Structureal characteristics

SEM image of the pore structure of the Ti-based getter is shown in Fig.1.

The Ti-based getter is a type of porous netlike structure. The presence of Mo powder is found to give the porous getter a high mechanical adherence due to some bonding formed with the other component at the sintering conditions. Thus the porosity and mechanical strength are guaranteed. A more precise study of the porosity of the getter material has been made by using a PBR bubble porosity pervasion method. The result is about 40 percent of the volume of the getter and the pore size present in the getter mass covers the range from 0.6 μm to 1.96μm. The average pore size is about 1.2 μ m. High specific surface area of the new getter is proved by the usual BET technique with Ar as the test gas, the value is up to 4000 cm^2/g, which is almost equal to the value of the original Ti powder before sintering. At high temperatures the sorbed rate limiting step is the bulk diffusion or the grain boundary diffusion depending on the conditions prevailing. However, at lower temperature surface reactions alone are responsible for the gettering process[2]. As a result, it is virtual to improve the pumping property by increasing the specific surface

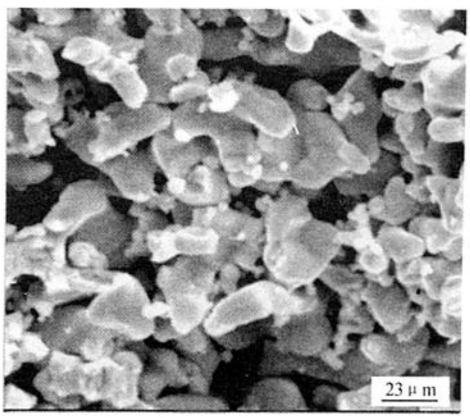

Fig. 1 SEM photomicrograph of the Ti-based getter

of the getter without increasing its physical dimensions. The relative high porosity and large surface area should allow good sorption characteristics at room temperature.

3.2 Sorption characteristics

The pumping tests of the new porous Ti-based getter for H_2 were performed after activation at 500℃ for 30 min, 600℃ for 30 min and 660℃ for 30 min.

The Ti-based getter was compared with the traditional same type getters St172 and St171, which were Zr-based getters. All of them were activated at 500℃×30 min, the results are shown in Table 1.

Table 1 Comparison of room temperature properties of three type getters

Getter materials	S_{10} 1)/($cm^3 \cdot s^{-1} \cdot cm^{-2}$)	Q_{60} 2)/($Pa \cdot cm^3 \cdot cm^{-2}$)
Ti-based getter	1418.6	997.1
St172	920	732.5
St171	421	346.5

Note: 1) S_{10} is the pumping speed at the tenth minute;

2) Q_{60} is the sorbed quantity after one hour

The results show that the pumping properties, including the characteristic pumping speed and the capacity after one hour , are much higher than traditional Zr-based getters under the same activation. When the Ti-based getter was activated below 500℃, weak pumping properties were present. Thus it can be concluded that the threshold of activation temperature is about 500℃.

Fig. 2 shows the time dependence of the pumping speed of Ti-based getter for H_2 at room temperature after different activation temperatures.

These results indicate that the pumping speeds of the new type Ti-based getter are very high at the beginning and decrease sharply in a few minutes, the curves are inclined to become steady later on. The pumping speeds measured after activation at high temperature are better than those at low temperature during the whole testing time. The results can be explained by the internal structure of the getter. The getter has large specific surface area to favor the accessibility of gas to be adsorbed to the surface of the getter while the gas diffusion in bulk is relatively weak. Since surface sorption dominates the initial step of gas absorption, the pumping speed begins to decrease greatly due to the fact that most of the surface of the getter is rapidly covered by the gas. When the surface sorption and the gas diffusion in bulk become almost balanceable, the trend of the curves become steady. As a result, this kind of getters can be applied in the vacuum tubes which release large amount of gas and need to be sorbed immediately.

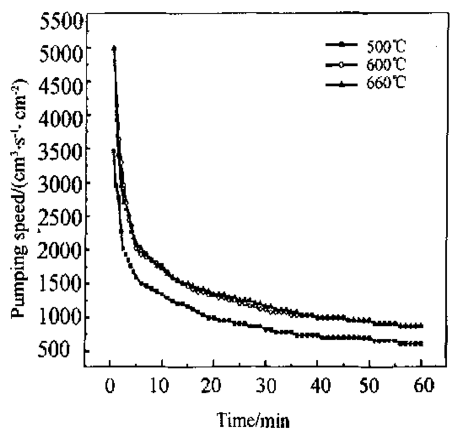

Fig. 2 Pumping speed for H_2 at room temperature after activations at different temperatures

In Fig. 3 the pumping speeds for H_2 at room temperature is plotted as a function of the sorbed quantity of the Ti-based getter after different activation temperatures.

It is shown that the pumping speeds can keep relatively high although a large amount of gas has been sorbed. The gettering properties can be improved by increasing the activation temperature from 500℃ to

600℃. When the getter is activated at high temperature, the bulk diffusion of the sorbed gas enhances, leading to the breakdown of the passivating layer, therefore more activated surface can be obtained to enhance the pumping speed and sorbed quantity. When the activation temperature increases to 660℃, the pumping property can no longer be improved, the curve is almost superposition with the curve measured under activation at 600℃, which may due to no further protective layer is destroyed when the activation has progressed to a certain extent. It can be concluded that the optimal activation temperature is around 600℃.

The Ti-based getter material, like all other non-evaporable getter materials, must be activated to enable it to function as a getter. The activation process must be carried out by heating under vacuum at a temperature, and for a time, such that from the surface of the getter material the thin passivation layer formed at room temperature during air exposure, is removed. To determine these conditions of activation, getters have been tested using different combinations of time and temperature. The result obtained for gettering characteristic of Ti-based getters for H_2 at room temperature after different activation processes is shown in Fig. 4.

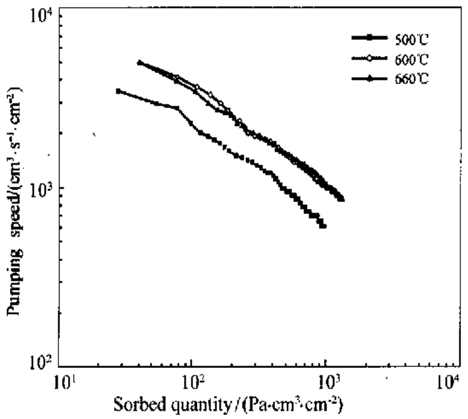

Fig. 3 Gettering characteristic for H_2 at room temperature after activations at different temperatures

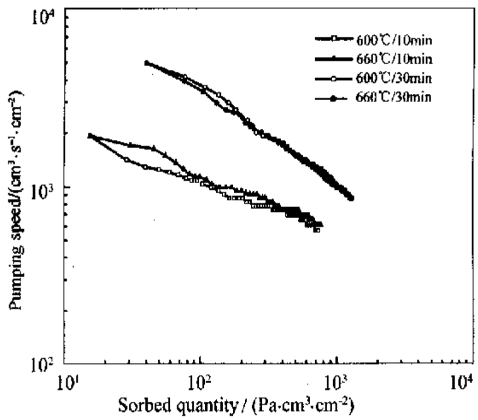

Fig. 4 Comparison of gettering characteristic for H_2 at room temperature after different activation processes

It can be observed that under the same activation time at 600℃ and 660℃, the activation temperatures have little effect on pumping properties. The pumping properties become higher by increasing the activation time. Prolonging the activation time, the activation will progress sufficiently to obtain more activated surface which is in favor of gettering properties. It can be concluded that the optimal activation condition of the new Ti-based getter is about 600℃ for 30 min.

4 Conclusions

(1) High porosity and large specific surface areas are in favor of the surface reaction to allow excellent pumping performances at room temperature, the Ti-based getter combines high pumping speeds and large capacity.

(2) The threshold of activation temperature is about 500℃ and the optimal activation condition is about 600℃ for 30 minutes.

(3) The new getter uses the sintering process under vacuum ambient to satisfy different configurations requirement by designing model.

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