ADSORPTION STUDY OF AL_2O_3 COATING ON THE PULSE SEPTUM MAGNET SURFACE

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Abstract

The surface of pulse septum magnet in HLS was specially coated with Al_2O_3 powder by plasma spraying. It was found with such magnet in the injection vacuum chamber the pressure was relatively low. To investigate the mechanics 20 sheets of coating were made (120×80 × 20mm^2), adsorption capacity was tested. Pore constitution was studied by N_2 isotherm method. Results indicated ultimate pressure reduced obviously from 6.4×10^{-5} Pa to 8.6×10^{-6} Pa. Coat adsorption capacity is about 3.68×10^{-4} Pa • l/cm^2. BET surface area is 201m^2/g, Coat is porous and pore is closed at one end. XPS results indicate there are hydroxyl, carboxyl etc on the surface, and they are most probably related with adsorption.

INTRODUCTION

HLS (HeFei Synchrotron Radiation Light Source) facility is a dedicated synchrotron radiation light source. It consists of a 200Mev LINAC and a 800Mev storage ring. The electron beam from transport line is injected into the storage ring by injection system which employs three kickers, a DC septum (D.S.) and a pulse septum (P.S.)[1].

D.S. magnet produce a deflection of 22.5° in the vertical direction and P.S. magnet produce a deflection of 6° in the horizontal direction for the coming beam. Electron beam goes into the vacuum chamber of the storage ring. Three kickers are fired and produce a localized bumped orbit close to the P.S. strip. The injection electron oscillate around the bumped orbit. In the following process, electron go inside the septum strip since the bumped orbit is contracted, so the injected beam run around the desired orbit.

It is important to develop a new material to reduce its outgassing rate so that the P.S. magnet could be directly mounted in the ultra-high vacuum chamber. Here a thin Al_2O_3 coating was sprayed on the surface of steel sheet and coil which composed the pulse septum magnet. With this coating in chamber pressure could reach less than 8×10^{-10}mbar after bakeout and pumped for 48 hour. Its electric insulation demonstrated good performance and magnet field undisturbed. Emittance of beam didn't get large. Injection efficiency increased[2][3].

In this paper some further study have been made to explore the mechanism of adsorption of Al_2O_3 coating.

EXPERIMENT AND TEST

Al_2O_3 coat was made. Its surface topography was observed. Its real surface area and adsorption capacity in vacuum chamber was tested. Adsorption species was investigated.

Coating Making

Al_2O_3 powder was sprayed on the steel sheet(120×80 ×0.6mm^3)by plasma spraying. Powder size is less than 0.065mm. Spraying parameters is listed in Table 1.

Table 1: Plasma Spray Parameters

Variable	Value
Ar flow rate	2000 l/h
H_2 flow rate	200 l/h
N_2 flow rate	250 l/h
Current	600A
Voltage	65V
Spray Distance	170mm
Spray angle	90°

Coating surface topography was observed by SEM (KYKY 1000B)(see in Fig.1)

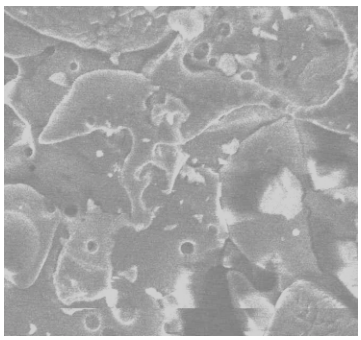

Figure 1: Al_2O_3 coating topography(×1000)

Obviously coating consists of numerous flat granules with many pores and gaps. Al_2O_3 granule is heated in the plasma flame and nearly in fusing state. It strikes towards base steel sheet so heavily that it became flat. Because granules are different in size, moment, fusing level, so their area, shape, thickness etc. are not alike. If two adjacent granule don't solidify simultaneously, a gap would be formed. The pores arise because impurity and oxide burning, oxide film bursting [4].

XRD

XRD results showed that powder Al_2O_3 is α phase, Al_2O_3 coated is γ phase.

Specific Area and Pore Constitution

N_2 adsorption isotherm was tested to investigate the real surface area and pore constitution of coating and

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powder. Tests were undertaken on ASAP 2000 system。Results are shown in Fig.2-5.

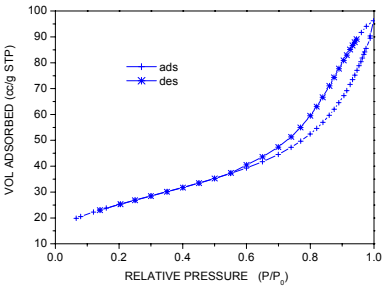


Figure 2: Al_2O_3 powder isotherm

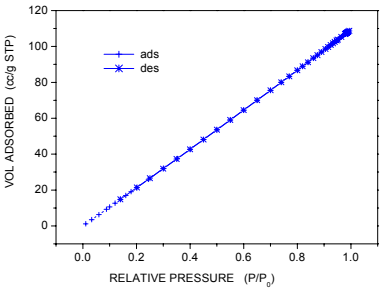


Figure 3: Al_2O_3 coating isotherm

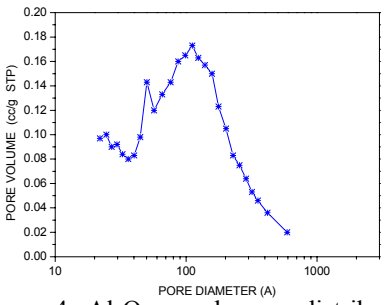


Figure 4: Al_2O_3 powder pore distribution

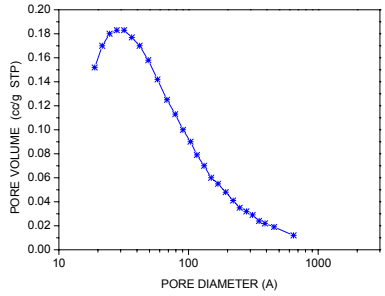


Figure 5: Al_2O_3 coating pore distribution

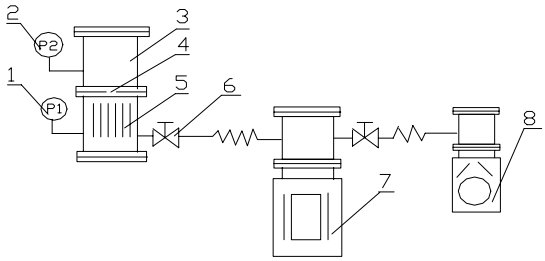
The specific surface area of powder Al_2O_3 is 91.1806m^2/g. It is clear in the isotherm curve that during adsorption process adsorbed volume increase slowly from p/p_0=0~0.65, but rises rapidly from p/p_0=0.65~1.0. No saturation appears up to P/P_0=1. During desorption process adsorbed volume decrease slower than counterpart during adsorption (see Fig.2). It is deduced that pore is of several shape: cone shaped or dual-cone shaped open at both-end, wedge-like open at four sides. Pores are existed among particles, formed by sloping face of particles. Multi-layer adsorption and capillary condensation occurs, interacting force between adsorbate and adsorbent is relatively fierce. In the pore distribution curve more than one peak are appeared. The highest peak is at between 100~110A, the second is about at 60A (see Fig 4). Surface area of pores which is larger than 50A accounts for 49.98% of all.

The specific surface area of coating Al_2O_3 is 201m^2/g. Both adsorption amount and desorption amount are both linear with pressure. No hysterisis appears (see Fig.3). It is deduced that the pore is cylinder-like, cone-like or wedge-like closed at one end. Multi-layer adsorption and capillary condensation also occur. Only one peak appears between 20~30A in the pore size distribution curve (see Fig.5). Surface area of pores which is larger than 50A accounts for 25.84% of all[5][6].

It is clearly demonstrated that from powder Al_2O_3 to Al_2O_3 coated real surface enlarge, pore diameter shrank and pore shape was changed completely.

Adsorption Capacity

Adsorption capacity of coating was tested quantitatively and quantitatively. The test system scheme is shown in Fig.6. 20 sheets of steel coated with Al_2O_3 (~3,840cm^2) was put directly into a standard test dome. Orifice diameter $d = 2\text{mm}$, flow conductance $c = 0.34\text{l/s}$ (27℃,N_2). P_1, P_2 was measured by two B-A gauge (L-B Co.), which had been calibrated at an all-metal ultra-high vacuum calibration facility. One 200l/s SIP was used to obtain ultimate pressure, One 110l/s TMP unit was used to obtain backing pressure.



1,2 B-A gauge 3 test dome 4 orifice 5 coating sheet
6 gate valve 7 SIP 8 TMP unit
Figure 6: test system sketch

Total leakage is less than 5×10^{-9} mbar • l/s. Chamber was baked at 250℃ for 24hrs then cooled down to room temperature. P_1,P_2 was recorded. Gate valve was not shut off until ultimate pressure was reached.

Adsorption capacity

$$\Delta Q = C(P_1 - P_2) = C(P_{10} - P_{20})$$

$$G = \int_0^T \Delta Q dt$$

P_{10}, P_{20} ---- background pressure.

The results are shown in Fig6,7.

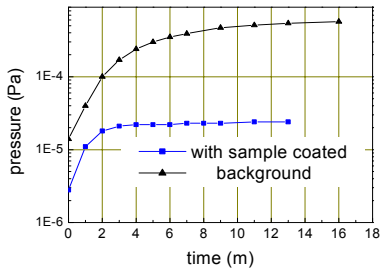


Figure 6: Pressure variation after gate valve shut off

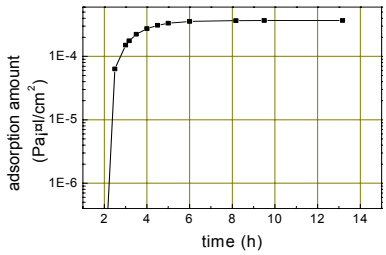


Figure 7: Al_2O_3 coating adsorption amount

With sample coated in the chamber ultimate pressure could reach 8.6×10^{-6}Pa, while background ultimate pressure is 6.4×10^{-5}Pa. Adsorption began to occur during cool-down process. In a few hours it attain saturation. Turn off the pump chamber could maintain certain pressure lower than background under the same condition. It means adsorption and desorption keep balance and coating has some absorbing capacity. It also imply the outgassing rate of coating is smaller than stainless steel.

XPS

XPS (X-Ray Photoelectron Spectroscopy) was used to investigate the adsorbate chemical state on surface. Excitation is Mg Kα , $h\nu$ =1253.6eV, analyzer energy CAE=20eV, step=0.05eV. Specimen was heated up to 250 ℃ in the vacuum and re-adsorption in the air. The surface spectrum and C1s spectrum are scanned (see in Fig8, 9).

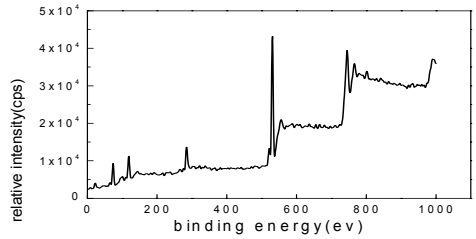


Figure.8: XPS of Al_2O_3 coating

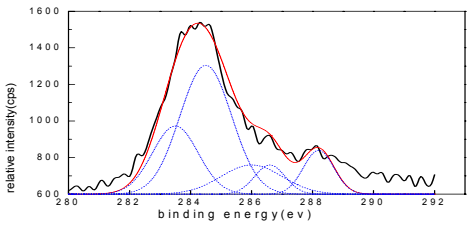


Figure.9: XPS of Al_2O_3 coating (C1s)

Content of elements is Al: 28.57%, O: 47.23%. 42.85% of O comes from Al_2O_3, the rest comes from adsorbate. There are several peaks at 283.5eV, 284.8eV 286Ev, 286.4eV and 288.1eV in C1s XPS spectrum. it is deduced kinds of chemical bonds existing on the surface as Al-O-C, C-H, –C–O, –C-OH, –C=O etc., which are most likely relate with the adsorption of CH_4,CO,CO_2 ,H_2O and other gases.

CONCLUSIONS

- The application of Al_2O_3 coating sprayed on the surface of magnet meterial brings some positive effects: difficulty of injection vacuum chamber design reducing, emittance of the beam no increasing, a good magnet performance, good vacuum condition and injection efficiency raising. The magnet has been successfully run for 10 years at NSRL.
- Due to particular forming process, Al_2O_3 coating have larger surface area than powder. It is beneficial to adsorption.
- The interaction between Al_2O_3 coating and gas is not only physical but also chemical adsorption.
- Further experiment and theory study on adsorption species is to be performed.

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