

Low-Melting Vanadate Glass Vaneetect Series VS-1298M

Takahiro Ikabata

New Materials Business Development Dept.,
Research & Innovation Promotion Headquarters

1 Abstract

Recently, housing thermal insulation regulations have been promoted, and Vacuum Insulation Glass (VIG) has attracted attention for heat insulation applications for windows. Low-melting Glass is used for VIG to achieve high airtightness and high reliability.

We have developed Low-melting Vanadate Glass, “VaneetectII” (400 °C bonding) and “VaneetectIII” (300 °C bonding). However, II has a high bonding temperature, and III is expensive because it contains silver. Therefore, we have developed “VS-1298M” (350 °C bonding), a silver-free vanadate glass with an extremely low melting point.

2 Characteristics of the Product

- Lowest melting point (350 °C) with no silver content.
- Sealing is possible in the atmosphere, inert gas, or vacuum.
- Good bonding with glass, ceramics, or metals.

3 Background of the Development

Currently, lead/bismuth glass that seals at 450 °C is the mainstream of Vacuum Insulation Glass (VIG), but there are always demands for lead-free glass that melts at a lower temperature. The addition of silver is effective for lowering the melting point of vanadate glass, but silver is expensive. Therefore, our goal was to develop glass that bonds at 350 °C without using silver. The issues to be overcome and our development guidelines are shown below.

- (1) No silver content. As silver is assumed to be incorporated into the glass in a cation state. We studied with alkali metals which are cations, instead of silver.
- (2) Glass temperature characteristics: Softening point is about 310 to 330 °C, and bonding is possible at 350 ± 10°C.
- (3) Crystallization temperature: Bonding temperature + 50 °C or above (400 °C or above). A high crystallization temperature is better, as low-melting glass loses adhesiveness when it crystallizes.
- (4) Coefficient of thermal expansion (CTE): Adjustable. If the CTE of the low-melting glass differs from the CTE of the adherend, the reliability is reduced due to peeling or cracking.
- (5) Chemical reliability: HAST, acid-, alkali-immersion tests and airtightness shall be equivalent to lead glass or bismuth glass.

4 Technical Details

Hitachi Chemical developed “VP-1298M” low-melting glass. The developed product contains no silver and can be melted and bonded at 350 °C. The glass transition temperature T_g , softening point T_s , and crystallization temperature T_{cry} were measured by differential thermal analysis (DTA). **Table 1** shows the measurement results for the newly developed product and a conventional product that bonds at 400 °C. In addition, we performed button flow test (hold at target temperature for 30 minutes) on compressed pellets of glass powder. The test results are shown in **Figure 1**. The pellet started to soften at 320 °C and flowed at 350 °C.

Table 1 Temperature Characteristics of Low Melting Glass

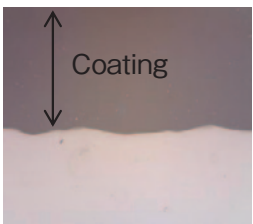
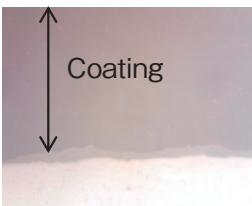
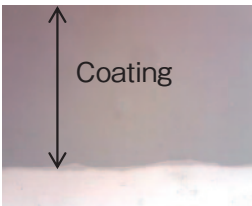
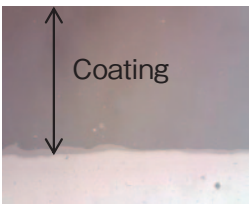
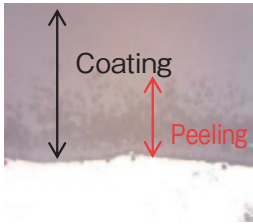
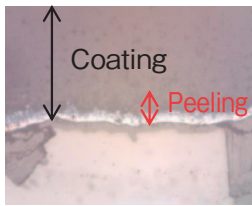
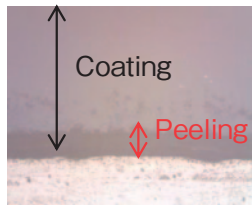
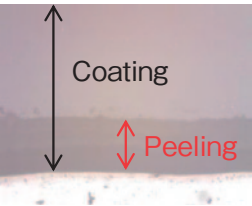
Sample		Developed product	Product that bonds at 400 °C
T_g	°C	261	312
T_s	°C	321	381
T_{cry}	°C	439	483



Figure 1 Button Flow Test of Glass Powder

The developed glass powder can be made into a paste by mixed with a thermal expansion filler, an organic solvent and binder resin. The glass paste can be applied in any place with a dispenser. We have developed “VS-1298M”, which has the same coefficient of thermal expansion as soda lime glass. Two soda lime glass (thickness 3 mm) were bonded at 350 °C with a bonding distance of 200 μm using “VS-1298M”. And HAST, saline immersion, acid immersion and alkali immersion tests were performed. The results are shown in **Table 2**. “VS-1298M” is a little peeled off in the reliability tests, it has good chemical reliability.

Table 2 Reliability Test Result of VS-1298M

Test	HAST	Saline immersion	Acid immersion	Alkali immersion
Conditions	120 °C, 0.199 MPa, 48 h	100 g/L NaCl, 35 °C, 1000 h	0.25 mol/L H ₂ SO ₄ , 50 °C, 2 h	0.5 mol/L NaOH, 50 °C, 2 h
Before test				
After test				

5 Future Business Development

- Provision of samples of the developed product and evaluation of their practicality