Gas Desorption of Inner Coating Materials Used for Cathode Ray Tube* —Effect of exposure time in the atmosphere on gas desorption—

Masao HASHIBA^{*1}, Yuko HIROHATA^{*1}, Tomoaki HINO^{*1},
Hidekazu YAMADA^{*2}, Akio KOIDE^{*2} and Yoshihiro KUBOTA^{*2}

^{*1}*Department of Nuclear Engineering, Hokkaido University, Sapporo, 060-8628, Japan*

^{*2}*Chemical Products Div., Hitachi Powdered Metals Co., Ltd.,
Tako-machi, Katori-gun, Chibaken, 289-2247, Japan*

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For two types of inner coating material of cathode ray tube (CRT) with different composition of water glass, the effect of air exposure in the atmosphere on the gas desorption was examined by using thermal desorption spectroscopy. The major outgassing species during the heating up to 773 K were CO₂ and H₂O in both the types. In the thermal desorption spectra of CO₂ and H₂O, a large desorption peak was observed at approximately 400 K. In both the materials, the amount of desorbed gases saturated at the exposure time of 1 h. Total amount of desorbed CO₂ and H₂O for the type with water glass content of (K₂O + Li₂O) · 3.8SiO₂ · 4H₂O was approximately a half of that for the type with water glass content of K₂O · 3.8SiO₂ · 4H₂O. The surface morphology of the former type showed fine structure compared with that of the latter type. If the former type is used for the inner coating material, the manufacturing process of CRT may be shortened compared with that of the latter type.

1. Introduction

A large cathode ray tube (CRT) for color television with a high performance has been still now developed. In order to lengthen the lifetime of CRT, the residual gas in the CRT has to be reduced for a depletion of the cathode to be suppressed. In a manufacturing process, the baking treatment has been conducted in the atmosphere and followed by in a vacuum. The maximum baking temperature is taken 673 K. After the baking in vacuum, residual gases such as H₂ and H₂O are gettered by flashing Ba metal¹⁾. Major gas source of the CRT is the inner coating material²⁾. Carbon based material has been used as the inner coatings to control the electrical conductivity at the surface. The electrostatic potential profile and electrical insulation depend on the conductivity. The composition of the inner coatings has been empirically determined so far. We studied on the gas desorption and adsorption properties for inner coating materials with different composition³⁻⁸⁾.

In this study, we examined the effect of air exposure time on gas desorption of inner coating material with different water glass composition, us-

ing a thermal desorption spectroscopy (TDS). Since the structure of the coating film depends on the composition of the inner coating material³⁻⁶⁾, the surface morphology was also examined by scanning electron microscope (SEM) and atomic force microscope (AFM).

2. Experiment

Inner coating material consisted of carbon powder with alkaline sol mixture, TiO₂ powder and water glass. Two types of inner coating material were prepared by using different water glass. The mass ratio of water glass were kept 32 wt.%. Two types of water glass, i.e., type A and type (A + B), were employed. The composition of water glass of type A was K₂O · 3.8SiO₂ · 4H₂O and that of type (A + B) was (K₂O + Li₂O) · 3.8SiO₂ · 4H₂O. Inner coating material was coated on the entire surface of 304 stainless steel plate with size of 20 mm × 60 mm × 0.05 mm by using a brush. The coating thickness was 3–5 μm. The weight of the inner coating was 11–18 mg. After the coating, the sample was dried at room temperature for 1 h. And then, the sample was heated at 723 K for 1 h in the atmosphere. After the heat treatment, the sample was exposed to the atmosphere with humidity 50% at 303 K. The exposure time was changed from 10 min to 24 h. In

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order to measure the absorption amount of gas, the sample was placed in a TDS apparatus⁸⁾. The TDS chamber was evacuated by a turbomolecular pump (TMP) and a rotary pump (RP). The ultimate pressure was 3×10^{-5} Pa. The total and partial pressures were monitored by a B-A gauge and a quadrupole mass spectrometer (QMS), respectively. The effective pumping speed was measured using a standard leak. The effective pumping speeds for CO₂ and H₂O were estimated as 0.044 m³/s and 0.055 m³/s, respectively. The sample was resistively heated from RT (296 K) to 773 K in the vacuum with a heating rate of 0.167 K/s. During the TDS measurement the sample temperature was monitored by a thermocouple spot-welded on the sample.

3. Results

Figure 1 shows the SEM photographs of both the samples of type A and type (A + B). No significant difference in the surface morphology between the them was observed. However, many small cracks were observed in the (A + B) type. We also observed the surface morphology by using AFM (**Fig.**

2). The surface of the (A + B) type had fine structure compared with that of A type sample. In addition, the effective surface area of the (A + B) type was approximately double of that of the A type⁸⁾. The observation by AFM is roughly consistent with that of the effective surface area. Thus, it is regarded that the (A + B) type is more porous than that of the A type.

In our previous study, it was found that the amounts of gas desorption and absorption significantly depended on the composition of water glass³⁻⁶⁾. In addition, the surface morphology and effective surface area of the inner coating material also depended on the composition of water glass³⁻⁶⁾.

Figure 3 shows the thermal desorption spectra of the A type after air exposure for 10 min (a) and 30 min (b). The major gas species desorbed from the sample were H₂O and CO₂. In the TDS spectra of H₂O and CO₂, a large desorption peak was observed at approximately 400 K. The spectrum of H₂O had a broad peak, although that of CO₂ was sharp. In the spectrum of CO₂, a second peak was

Fig. 1 SEM photographs for A type and (A + B) type.

Fig. 2 AFM photographs of A type and (A + B) type.

Fig. 3 Thermal desorption spectra of H_2O and CO_2 of A type after air exposure with exposure time for 10 min (a), and for 30 min (b).

Fig. 4 Thermal desorption spectra of H_2O and CO_2 for A type (a) and (A+B) type (b) after air exposure for 30 min at 303 K.

observed at 710–760 K. The amount of desorbed H_2O and CO_2 increased with the exposure time in the atmosphere. In the (A+B) type, the spectra were roughly similar with the case of the A type, although the desorption amount was small.

Figure 4 shows the thermal desorption spectra of H_2O and CO_2 for the A type (a) and (A+B) type (b) after air exposure for 30 min. The desorption amounts of H_2O and CO_2 for the (A+B) type were smaller than those of A type. The second peak of CO_2 spectrum in the (A+B) type appeared at a lower temperature region, 580 K. The amounts of desorbed gases were obtained by integrating for the desorption rate with respect to the heating time up to 773 K. **Figure 5** shows the desorption amounts of H_2O and CO_2 as a function of the exposure time in the atmosphere. The desorption amounts of H_2O and CO_2 increased with the exposure time in both the types, and saturated for 1 h. In the manufacture process, the air exposure time after baking is taken shorter than 1 h. In this case, the desorption amount of the (A+B) type is approximately a half of that of A type. The viscosity of the water glass used for the (A+B) type is smaller than that of A type. The gas absorption mainly takes place in the

Fig. 5 Desorption amounts of H_2O and CO_2 versus air exposure time.

carbon powders. Hence, in the case of the (A+B) type, the surface of carbon powders might be sufficiently coated with the water glass. This may be a reason that the gas absorption amount of the (A+B) type was smaller.

4. Conclusion

For two types of inner coating material with different composition of water glass, the effect of

air exposure time in the atmosphere on the gas desorption was examined using TDS.

Major gas species desorbed from both the types were H_2O and CO_2 . The desorption amount of gases saturated at exposure time of 1 h in both the types. The desorption amount of gases for the (A + B) type was a half of that for the A type. The surface morphology of the (A + B) type had fine structure, compared with that of the A type.

If the (A + B) type is employed in the manufacture process, the baking temperature can be reduced and the pumping time can be shortened, compared with a case of the A type.

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