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(57)

Development of Sealants and Adhesives for HDDs

Introduction

Hard-disk drives (HDDs) have played an essential role in recent progress in information technology and digitalization. More than 200 million hard disks are produced each year as storage devices for both personal and mainframe computers. These hard disks offer high capacity, high-speed access, and cost-effective performance. Three Bond develops anaerobic sealants, epoxy resins, ultraviolet-curing resins, and conductive resins that have low-outgas characteristics, and they are used as sealants and adhesives in current HDDs. This issue introduces technologies to produce low-outgas sealant and adhesive, their capabilities, and methods to measure outgas.

HDD is a sealed storage device. Therefore, if any corrosive, volatile gases are generated inside the device, it may impact device performances. HDDs are magnetic storage devices that incorporate magnetic heads to record signals to a recording film (or medium) deposited upon on smooth aluminum or glass disks rotating at some 5,000 to 10,000 revolutions per minute. Clearance between the head and the surface of the medium is on the order of several tens of nanometers. Therefore, any foreign objects adhere between the head and the disk may cause a major damage. For this reason, major imperatives in HDD development are to protect the devices from penetration of small dust and humidity, and reduce the generation of volatile gasses from parts and adhesives used to assemble the devices. These adhesives used for this purpose have numerous requirements other than low outgas levels, including special functionality on workability, productivity, quality control and supply management of the products. All Three Bond groups in domestic and overseas development, sales, and manufacturing plants work in concert to ensure timely product development and stable product supply that fulfills these requirements.

In the rest of this document, Three Bond is abbreviated TB.

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Sealants and adhesives used in hard disks

Figure 1 shows an exploded view of a HDD and the Three Bond sealants and adhesives used in a HDD. Three Bond offers a variety of sealants and adhesives for hard disks.

1-1. Various sealants and adhesives for hard

Many Three Bond products are used in hard disks. Ultraviolet-curing sealants are used for the top cover of the hard disk; anaerobic resins and epoxy resins are used in the spindle motor to fix the bearing and shaft and to keep the magnet in place. Ultraviolet-curing resins and conductive resins are used in the magnetic head and suspension. Epoxy resins are used in the voice coil motor (VCM) to fix the coil and the magnet.

Three Bond develops and offers various sealants and adhesives specifically matched to each region of HDD assemblies, aiming to minimize total outgas generated from sealants and adhesives.

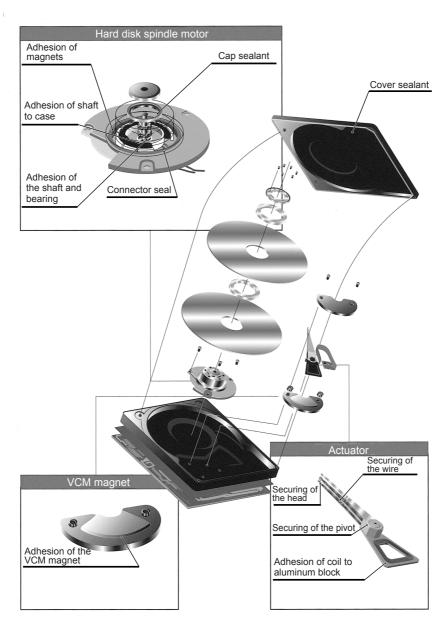


Figure 1: Exploded view of HDD and sealants and adhesives used

1-2. Understanding outgas that sealants and adhesives generate 1)

Outgas generated from sealants and adhesives are volatile organic or inorganic gases of low molecular weights up to around 300, generated during curing time or from cured objects after curing completed. Reducing outgas from sealants and adhesives is specifically required inside HDDs.

Figure 2 shows the thermal mass change when a cured mass of TB3057H ultraviolet-curing resin is heated to 500°C. Increasing the temperature gradually decreases mass. Thermal decomposition of the cured object starts at approximately 300°C. The temperature inside HDDs goes up to approximately 100°C. Specifically the thermal mass decreased before starting thermal decomposition equals to the outgas.

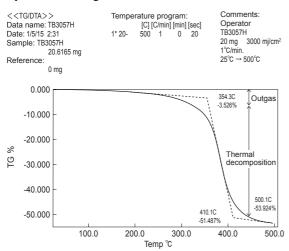


Figure 2: Thermally-induced mass change rate during heating of TB3057H

1-3. Outgas components

The volume and components of outgas differ from adhesive to adhesive. Major outgas components include the moisture, reduction objects of curing agents, polymerization inhibitors, un-reacted monomers, organic metal catalysts, organic acids, and impurities in raw materials such as organic solvents that are contained in sealants and adhesives. The outgas components that pose serious failures to HDDs are those with strong adhesive and corrosive properties.

2. Methods of measuring outgas

Chemical analyzers such as GC-MS (gas chromatograph-mass spectrometer) and TGA (thermo-gravimetric analyzer) are used to measure the outgas generated from cured adhesives. In particular, GC-MS, which combines gas chromatography and mass spectroscopy, is used in

many industries as standard equipment for qualitative and quantitative outgas analysis. GC-MS has recently begun to be used to analyze clean-room contaminants and to detect endocrine disruptors.

2-1. Qualitative and quantitative determination

The outgas components generated by sealants and adhesives are measured to determine the volume of gas generated (quantitative determination) and to identify the components (qualitative determination). It is required to know the volume of generated gas and its components from sealants and adhesives. The gas components must also be scrutinized whether they are corrosive, or not.

2-2. Measurement of thermal mass reduction using TGA

TGA is used to measure changes in the mass of sealants and adhesives during application of heat. TGA equipment can measure the absolute volume of the moisture, and inorganic and organic gas generated from sealants and adhesives under specific heating conditions. This measurement clarifies the relationship between changes in mass and time or temperatures. However, it cannot identify the components of the generated gas or the proportions of each component in the gas generated. Figure 3 shows the thermal mass changes on TB3057H caused by continuous application of heat of 80°C and 120°C. Outgas evaluation is typically performed with continuous measurement at an optional temperature between 80°C and 150°C. The change in thermal mass at the temperature is measured and the volume of outgas is determined.

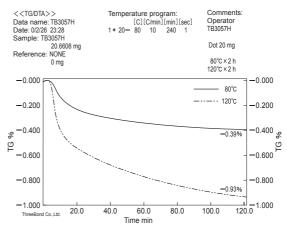


Figure 3: Thermal mass change rate during continuous application of heat of 80°C and 120°C on TB3057H

2-3. Qualitative and quantitative determination using GC-MS ²⁾

GC-MS can perform quantitative and qualitative determination of organic gases with molecular weights approximately ranging from 30 to 500. This equipment also provides various methods for extracting outgas, including the head space method and the dynamic head space (DHS) method. Since the DHS method uses a constant flow of inert gas in the sample chamber and adsorbs the generated outgas to an adsorbent, it enables analysis with high sensitivity. Figure 4 is a schematic diagram of the GC-MS used by Three Bond. Table 1 shows results of GC-MS measurement on TB3061H.

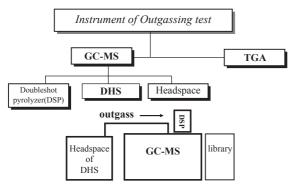


Figure 4: Schematic diagram of outgas measurement equipment

3. Anaerobic sealants and ultraviolet-curing resins

3-1. Anaerobic sealants and ultraviolet-curing resins for HDDs ³⁾

Anaerobic sealants are one-part non-solvent adhesives that react at room temperature in small gaps between metals. Therefore, this characteristic is particularly well suited to surface adhesion and fitting adhesion applications such as fixing the shaft and bearing. Other one-part non-solvent adhesives include ultraviolet-curing resins, which cure rapidly when irradiated with ultraviolet light.

2-4. Evaluation of measurements

Quantitative values and qualitative components measured are varied with measurement equipment, curing conditions, shape and surface area of the cured object. Also, measurements of the adhesive itself are different with ones of the adhesive used in actual assembly. These factors must be considered when evaluating measurement results. Also, diversified measurements ensure certain evaluation.

Table 1: GC-MS measurement results for TB3061H

Outgassing of ThreeBond 3061H (Curing condition 3000 mJ/cm²) GC-MS/DHS	%
Acetone	2.36
d-Glycero-d-ido-heptose	4.65
butanoic acid, 2-hydroxyethyl ester	10.92
Benzene,(1-methylethyl)	10.72
2-propenoic acid, 2-hydroxyethyl ester	5.5
Benzaldehyde	0.93
Alpha-methylstyrene	1.03
Acetophenone	9.00
Benzenmethanol alpha, alpha, -dimethyl	22.85
2-Furanmethanol, tetrahydro-, acetate	12.07
benzene,1,4-bis(1-methylethenyl)-	10.59
propandoic acid,(benzoylhydrazino)hydroxyl-,	5.34
dimetyl ester	
other	4.13

These sealants and adhesives are ideal for fixing parts and for sealing against oil and dust. Sealants and adhesives are primarily classified as acrylic- or epoxy-based resins. Both anaerobic sealants and ultraviolet-curing resins start the curing reaction by resolving curing catalysts in curing time. Part of the resolution material volatilizes from the cured object and forms the outgas. Table 2 lists the anaerobic sealants and ultraviolet-curing resins used in HDDs.

Table 2: Anaerobic sealants and ultraviolet-curing resins for hard disks

Product name			ThreeBond 1360D	ThreeBond 1372D	ThreeBond 3057H	ThreeBond 3059D	ThreeBond 3061H
			Anaerobic sealant	Anaerobic sealant	Ultraviolet-curing resin	Ultraviolet-curing resin	Ultraviolet-curing resin
			Anaerobic Sealant	UV curing added	UV and heat curing	UV curing only	UV and anaerobic curing
Color and appe	arance			Green	Blue	Creamy white	Dark Blue
Viscosity		Pa∙s (cP)	0.90 (900)	0.11 (110)	12 (12000)	76 (76000)	8 (8000)
Specific gravity			-	-	1.14	1.18	1.14
Hardness			-	-	D 85	D 88	D 65
Compressed	Iron	MPa {kgf/cm²}	37.1 (371)	33 (336)	-	-	-
shearing	Stainless steel	MPa {kgf/cm²}	20.7 (207)	31 (305)	-	-	-
strength	Aluminum	MPa {kgf/cm²}	28.1 (281)	27 (275)	-	-	-
Shearing	Glass/glass	MPa {kgf/cm ² }	-	-	20.5 (205)SUS/SUS *3	8.0 (80)	8.8 (88)
adhesive strength	Acrylic/acrylic	MPa {kgf/cm²}	-	-	5.2(52) SUS/Acrylic	3.0 (30)	7.0 (70)
Glass transition	point (DMA tan δ)	°C%	165	147	85.8	82	35
Rate of therma	mass change	×10 ⁻⁶ / °C	-3 ^{*1}	-35 ^{*1}	-0.6 *2	-0.6 *2	-0.9 *2
Linear expansion	on coefficient		81.9	80.5	46.8	-	210
Applications			Fixing of spindle	Fixing of spindle	Sealant for spindle motor	Fixing wires	Sealant for spindle motor
			motor shafts	motor shafts	caps	High viscosity	caps
			Primer TB1390K	Primer TB1390E, F	Homogeneous sealant	Ì	Heterogeneous sealant

1 Uncured resin at 80°C after 16 hours

Cured resin at 120°C after 2 hours

Curing conditions: 100°C × 0.5 hours

3-2. A new anaerobic sealant: TB1360D

The TB1360D anaerobic sealant was developed for fitting adhesion of the shaft and the bearing of the spindle motor. This material offers a high glass transition temperature of 165°C and low volatile content in the uncured object (liquid). For shaft adhesion of the spindle motor, provisions against heat are particularly important while the disk is spinning; if the shaft shifts even slightly (on the order of sub-microns), the disk on the shaft (particularly at outer perimeter) will inevitably shift significantly (on the order of microns). The resin used here must be heat-resistant; in other words, the resin must have a high glass transition temperature. Figure 5 shows the heated strength of TB1360D. Figure 6 shows the thermal degradation of strength on TB1360D and TB1373B. TB1373B is an older product. TB1360D clearly offers performance. Here, excess anaerobic sealant in the fitting adhesion does not cure with oxygen and releases significant volumes of outgas. TB1360D significantly reduces outgas emitted from excess sealant, compared to older sealants. Due to their potential causes to harm HDD components, TB1360D also uses no organic sulfur compounds, however, used in combination with a special TB1390K primer, it is achieving anaerobic curing properties comparable to older products.

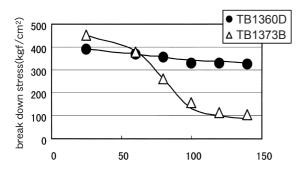


Figure 5: Comparison of hot strength between TB1360D and TB1373B

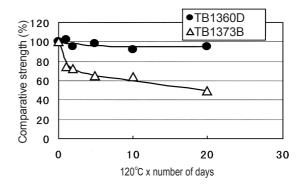


Figure 6: Comparison of thermal degradation between TB1360D and TB1373B

3-3. Ultraviolet-curing resin TB3061H

Ultraviolet-curing resin TB3061H is a one-part non-solvent acrylic-based adhesive with anaerobic curing properties. Developed as a cap sealant for the spindle motor. TB3061H offers excellent ultraviolet-curing properties and adhesion capabilities with metals and plastics. Similar to TB1360D, it contains no organic sulfur or organic tin compounds. Figure 7 compares the reaction ratios of TB3061H and an old product. The acrylyl group reaction ratio is measured by real-time FT-IR (Fourier transform infrared spectroscopy). The figure clearly shows that TB3061H completes the reaction rapidly.

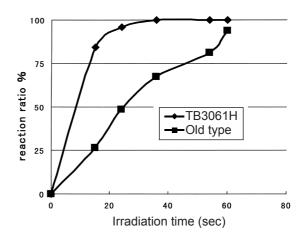


Figure 7: Ultraviolet irradiation time and reaction ratios of TB3061H and an older product

4. Epoxy resins

Epoxy resins for hard disks are classified as one-part and two-part resins. Epoxy resins generate less outgas than anaerobic sealants and ultraviolet-curing resins. This is due to the curing mechanism of epoxy resins, which does not

generate resolution objects. The cured objects of epoxy resins are also hard (due to the three dimensional mesh structure), and have the capability to suppress the volatilization of gas components with low molecular weights contained in them. Table 3 lists the epoxy resins for HDDs.

Product name		ThreeBond 2202	ThreeBond 2270C	ThreeBond 2087F		ThreeBond 2087G		Product under development	
				Primary agent	Curing agent	Primary agent	Curing agent	Primary agent	Curing agent
Color		Black	Black	Pink	Blue	Pale blue	Red	Transparent yellow	Transparent deep blue green
Viscosity	Pa∙s (cP)	13 (130)	70 (700)	13 (130)	4.4 (44)	13 (130)	5 (50)	2.5 (25)	3 (30)
Specific gravity		1.14	1.95	1.17	1.10	1.20	1.25	1.19	1.00
Standard curing condition		80°C × 20 minutes	100° C × 40 minutes 120° C × 30 minutes 150° C × 20 minutes	60°C × 3 hours 80°C × 1 hours 80°C × 10 minutes 80°C × 10 minutes		90°C × 1 hour			
Working time	minutes			25-30 (when mixing 100 g)		80 (when mixing 100 g)		240 (when mixing 100 g)	
Shearing adhesive strength	MPa {kgf/cm²}	10.0 (102)	21.6 (220)	28.4 (290)		11.8 (120)		12.9(131)	
Peeling adhesive strength	N/m {kgf/25mm}	160 (0.4)	2100 (5.4)	589 (1.5)					
Glass transition point	°C	105	140	8	5	145		130	
Thermal mass change rate	%			At or below 0.001 At or below 0.001		ow 0.001	At or below 0.001		
Linear expansion coefficient	×10 ⁻⁶ / °C	74	41	72		72 60		60	
Applications		Bonding spindle motor rotor magnets Bonding general hard disk parts	Bonding VCM (Coil and arm)	Bonding spindle motor rotor magnets Bonding general hard disk parts		rotor magnets Bonding general hard rotor magnets Bonding general hard		For FDB oil sealing	

Table 3: List of epoxy resins for hard disks

4-1. Two-part epoxy resins TB2087F and TB2087G

A two-part epoxy resin consists of a main agent and a curing agent. These components are mixed in the appropriate ratios and heated to cure. TB2087F and TB2087G are designed for use to fix the magnet in the spindle motor.

Offering lesser changes in viscosity than older adhesives, (longer working time) after mixing at room temperature, TB2087F and TB2087G are also able to cure at lower temperatures. Figure 8 shows appearance in the curing of TB2087G measured by Rheometer. TB2087G shows small viscosity change around room temperature, but after application of heat, it starts curing reaction in a short time.

In addition to the products above, we are considering to market products of two-part oil-resistant epoxy resin designed to seal oil within fluid dynamic bearing (FDB) motors. Compounding and mixing are crucial for two-part epoxy resins, since errors in the area may leave uncured portion of the resin. To prevent this, Three Bond's epoxy resins use different colors for the primary agent and the curing agent, enabling easily visualize the mixing state.

4-2. One-part epoxy resin TB2270C

One-part epoxy resins are one-part non-solvent resins that contain powdered curing agents dispersed in the main agent. They do not have a working time as defined for two-part resins. TB2270C is used to bond the coil and the arm of the VCM. TB2270C offers high thermal conductivity

and excellent adhesive strength with metals. The cured objects are hard and strong and do not affect VCM operations. Table 4 shows the results of the adhesive strength test after the PCT. Since one-part epoxy resins use powdered curing agents, they are unsuitable for fitting adhesion applications such as fixing the shaft and bearing. Since the curing agent would partially penetrate into the fitting area as inserting the shaft, which would leave uncured resin.

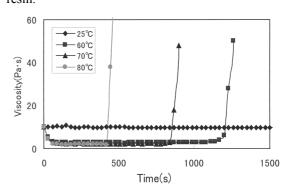


Figure 8: Viscosity changes in TB2087G by time at various temperatures, as measured by a Rheometer

Table 4: Adhesive strength of TB2270B after PCT

Items	ThreeBond 2270C			Competing product
Curing	120°C	120°C	120°C	130°C × 50min
conditions	× 40min	× 40min	× 20min	+155°C × 120min
Blank	41 MPa	39 MPa	44 MPa	40 MPa
After PCT	21 MPa	21 MPa	23 MPa	Peeled directly after

*PCT conditions: 121°C, 2 atm, after 12 hours

Conductive resin

A conductive resin consists of a binder such as an epoxy resin and a conductive powder (conductive filler) such as silver powder. The conductive filler, constituting 80 to 90% of total weight is dispersed throughout the binder. Conductivity emerges as the conductive filler objects become closer when

volume shrinks during binder curing. The high velocity rotations of hard disk generate static electricity, which may cause significant damage on the magnetic heads and medium. Conductive resins are used to remove this static electricity. Figure 5 illustrates the properties of conductive resins for HDDs.

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Product name		ThreeBond 3380E		ThreeBond 3305B
		Primary agent	Curing agent	
Color and appearance		Silver	Gray	Burnt umber
Viscosity	Pa∙s (P)	31 (310)	2.9 (29)	22 (220)
Specific gravity		3.7	3.2	1.8
Standard curing condition		60°C × 3	3 hours	120°C × 30 minutes
Working time	Minutes	240 (*1)		-
Shearing adhesive strength	MPa {kgf/cm²}	19.5 (198) (*2)		16.9 (172) (*2)
Peeling adhesive strength	N/m {kgf/25mm}	-		2.7 (6.7)
Glass transition point	°C	-		17
Thermal mass change rate	%	0.09 (*3)		0.10 (*4)
Linear expansion coefficient	×10 ⁻⁶ /°C	- ` ´		-
Applications		Cap seal		For assembling hard disk magnetic read/write heads

^{*1:} Time after mixing required for viscosity to double

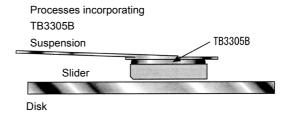
5-1. Two-part non-solvent conductive resin TB3380B

TB3380B conductive resin uses a two-part epoxy resin (non-solvent) as binder. Both the primary agent and the curing agent contain dispersed silver powder conductive filler. TB3380B is used to seal the spindle motor and drain static electricity. TB3380B forms flexible cured objects that accommodate heat cycles with stable volume resistance values.

5-2. TB3305B ultraviolet-curing conductive resin

The TB3305B ultraviolet-curing conductive resin uses a one-part ultraviolet-curing resin as binder and silver powder (spherical powder of 10 microns) as conductive filler. TB3305B is used to bond the slider and suspension and to remove static electricity from these parts. To date, two types of resins have been used to meet this requirement: an ultraviolet curing resin for adhesion and a conductive resin to remove static electricity. TB3305B simplifies the process by serving both roles. TB3305B provides temporary binding when irradiated with ultraviolet light, complete curing of shadow areas with heat and enables vertical conductivity (removal of static electricity) over small vertical gaps of 10 microns or less. The cured objects are flexible, create minimal stress when

curing, and do not increase strain on the magnetic heads. Figure 9 illustrates an application sample of TB3305B.



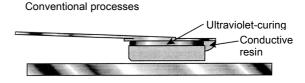


Figure 9: Illustrated example of TB3305B application

^{*2: 2 \}phi ceramic chip/glass adhesive strength

^{*3:} Aging at 120°C for 1 hour

^{*4:} Aging at 120°C for 2 hours

6. Summary

The low outgas sealant and adhesive technologies discussed in this article have become essential for electronic devices other than HDDs. Low outgas requirements will continue to increase for sealants used in liquid crystals and EL display boards, used as conductive resins for securely positioning quartz oscillators, or used as adhesives around optical pickup lenses. We remain dedicated to advancing the state of the art in resin functionality and outgas characteristics.

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References

- 1) Storage Device Terminology, Nikkei BP
- 2) Tatsuo Nonaka, et al., Sumika Chemical Analysis Service, Ltd., Outgas analysis of materials, including clean room components, Clean Technology, Vol. 9, No. 2, Japan Industrial Publishing Co., Ltd.
- 3) Aki Kusuyama, Three Bond Co., Ltd., Anaerobic sealants in electric applications, Industrial Materials, Vol. 49, No. 5

