

Anaerobic Adhesive and Sealant

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Introduction

Screws constitute one of most important fastening-joining elements in assembling various components of machines and equipments in every industry. While screws are convenient and easy to assemble and disassemble, they have some demerits, such as loosening under some circumstances, and failure of sealing owing to clearance caused by tolerance.

Three Bond has developed many years ago anaerobic adhesive and sealant, Three Bond (to be abbreviated as TB hereinafter) #1300s to solve these problems chemically.

Recently, high efficiency application systems involving automatic applicators utilizing the fluidity of AAS have been developed, allowing to use AAS effectively without waste, to expand its usage steadily and to contribute to industries. This is very encouraging to us, who pursued its development.

This report concerns technical explanations on the background, reaction mechanism, characteristics, durability and problems to be solved of liquid AAS.

1. History and Background of AAS

Anaerobic adhesive and sealant first attracted attention in 1945, when Nordlander and Burnett of the G.E. (General Electric) laboratory had found that dimetacrylate with oxygen dissolved at higher temperatures hardened as being held between a microscope slide glass and a sheet of metal foil. They designated such substance “anaerobic permafил”.

“Anaerobic” is an adjective used in biology for organisms which can live on in the oxygen-less environment.

The commercialization of AAS started in 1955 in U.S.A. In Japan, the development of AAS was initiated in 1963, and the commercialization started in 1965.

AAS is defined by the Japan Adhesive Industry Society Standards (JAI-6, 1979) in the following way: “compounds which polymerize and harden when isolated from air between two adjacent faces, so as to be suitable for locking metal parts, sealing and fastening fitting”.

That is, AAS is adhesive of peculiar characteristics, while being liquid in the presence of air, rapidly curing by polymerization if cut off from air in the state of film in gap at a screw or a fitting.

Upon polymerization, AAS turns to resin of dense three-dimensional reticular structure, which has excellent anticorrosiveness, solvent resistance, thermal resistance, aging resistance. Since no solvent is involved, the polymerization produces little contraction, to be suitable for fixing and sealing fitting, pipe and flange.

Adhesive is distinguished from sealant in the following way: adhesive is simply to bond together, while sealant has both sealing and locking effects in addition to bonding. In the Japan Adhesive Industry Society Standards, where plane sealing and plane bonding are excluded, AAS is called “adhesive”.

Since the research and development at Three Bond concern a broad field including sealing, we use “adhesive and sealant” for this compound.

2. Reaction of Mechanism of AAS

2-1 Radicals

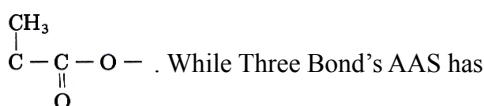
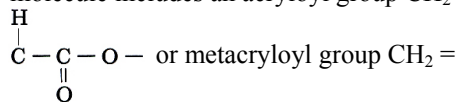
The AAS reaction is a sort of radical polymerization. Radical may be simply defined

as a chemical species, characterized by the presence of unpaired electron. A chemical bond is normally formed by a pair of electrons. Such a compound is dissociated in either of two ways:

- 1) The electron pair are shifted to one of two dissociation products, $A : A \rightarrow A^+ + B^-$. This process is called heterolysis, and dissociation products are ions.
- 2) Two electrons are distributed to each of two dissociation products, $A : B \rightarrow A\cdot + B\cdot$. This process is called heterolysis, and dissociation products are radicals.

2-2 Conditions of AAS

To be an AAS, it is essential that the molecule includes an acryloyl group $CH_2 =$



dimetacrylate with two metacryloyl groups as main component, there are AASs with one, three or more of these groups. Moreover, *organic peroxide is required, usually as a polymerization initiator.

The polymerization of AAS requires three conditions:

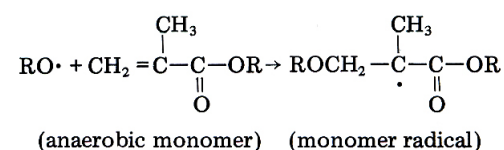
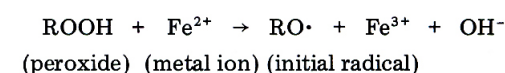
- 1) Isolation from air (oxygen)
- 2) Contact with metal
- 3) Appropriate temperature.

As for the temperature, the chemical reaction underlying the curing mechanism is extensively affected by temperature. It should be noted, therefore, that the curing rate is extremely retarded at 10°C or lower, and nearly null at 5°C or lower.

For 1) and 2), see the next subsection.

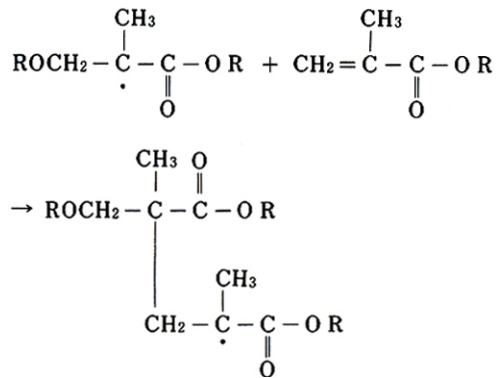
2-3 Reaction Mechanism

1) Initial Reaction

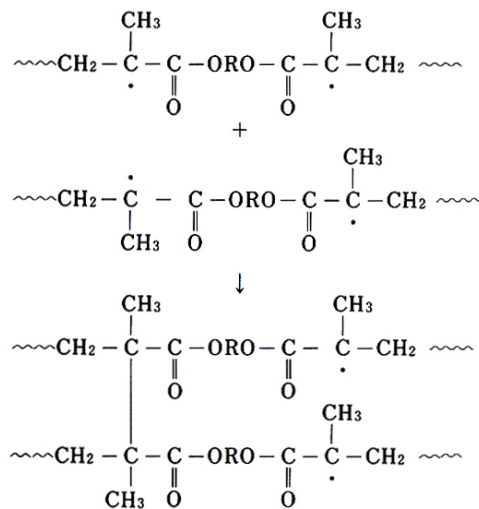


The reaction between peroxide and metal ion produces initial radical to start the polymerization.

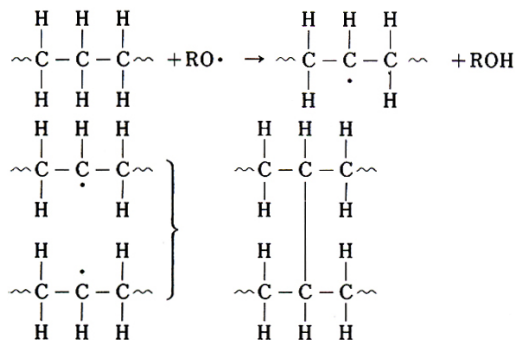
2) Growth Reaction



This reaction proceeds rapidly to make monomers polymerize. In case of dimetacrylate (as with tri- and tetra-metacrylate), cross-linkage may occur between polymers.

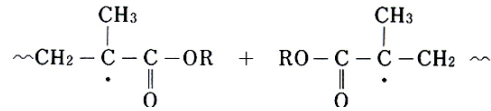


The cross-linkage may be caused through the growth reaction mentioned in the above, or through the dehydrogenative due to RO-radical derived from peroxide.



As a consequence of these complicated series of reactions, three-dimensional reticular structures are formed.

3) Stopping Reaction

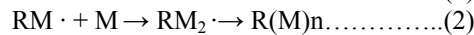
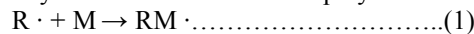


The reaction stops here, to finish curing.

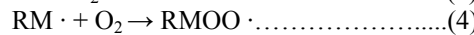
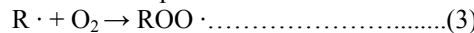
2-4 Role of Air (Oxygen)

AAS is based on the inhibition of radical polymerization by oxygen. Reactions described in 2-3 occur only if oxygen is excluded. While the reaction involving oxygen is not yet decisively elucidated, the following mechanism may be conceived.

In the presence of air, or within an AAS container, made from permeable polyethylene, peroxide is decomposed, by the action of polymerization accelerator or reducing agent, without metal ion, the release radicals, which naturally tend to make monomers polymerize.



However, as oxygen exists, the following reactions are anticipated.



where M: monomer, R·: initial radical, RM·: monomer radical, ROO· and RMOO·: peroxide radical (of lower activity), O₂: oxygen molecule.

The reaction rates are said to be: $k_{(1)} \doteq k_{(2)}$, $k_{(3)} \doteq k_{(4)}$, $k_{(2)}/k_{(4)} = 3 \times 10^{-5}$ and $k_{(5)}/\sqrt{k_{(6)}} = 1.39 \times 10^{-3}$.

As active initial radicals are little utilized for the growth reaction, and most of peroxide radicals ROO· turn to inert substance, no polymerization occurs.

While AAS includes polymerization inhibitors, its effect is far less than the inhibitive effect of oxygen.

3. Classification of Main ThreeBond AAS Products

Series	Usage	Division	Strength	Viscosity	TB Grade	Remarks
TB1300 series	Screw	Unremovable stud bolt	High	Low Medium	1 3 0 3 1 3 0 5	
		Removable, but requiring bond strength	Medium	Low Medium High	1 3 2 2 1 3 2 4 1 3 2 7	
		Frequently removed	Low	Low Medium	1 3 4 2 1 3 4 4	
		Heat-resistive	Medium	Medium	1 3 6 0	
		For stabilizing axial force (lubricated type)	Medium	Medium	1 3 7 4	
	Fitting	For smaller components	High	Low	1 3 7 3 B	
		For general purpose, heat resistive	High	Medium	1 3 7 5 B	
		For larger components	High	High	1 3 7 7 B	
		For larger components	High	Very high	1 3 7 9 B	
	Special Purpose	Back-impregnation	Medium	Very low	1 3 6 1 B	for filling-up of pinholes
Sealing for weld of tire wheel rim		Medium	Low	1 3 7 0 B	Spraying application possible	
Sealing for welch plug		Medium	High	1 3 8 6 Series	Thixotropic properties	
Standards	National standards					
	User's or special specifications					
TB1100 series	Sealing	For taper plug	Low	Paste	1 1 3 0	Tumbler application
		For taper plug, heat-resistive	Low	Paste	1 1 3 2	
		For general piping	Medium	Paste	1 1 1 0 B	
		For flange	Low	Paste	1 1 3 1	Screen printing

The characteristics of AAS will be described for various categories.

4. AAS for Screw

4-1 Characteristics of AAS for Screw

Type	Color	Viscosity C P S ±20% (25° C)	Break loose torque (kgf·cm) 10φ JIS-2 grade with mild steel bolt-nut		Curing Speed (20~25° C)		Main features and usage
			Break-loose torque	Prevailing torque	Time to usable strength	Time to ultimate strength	
TB1303	Green	150	300~350	250~350	30 min~2 hrs	24 hrs	High bonding strength, fast setting For fastening fittings
TB1305	Green	600	300~350	250~300	30 min~2 hrs	24hrs	High strength fastening of screws
TB1322	Red	150	100~150	250~300	1 hr~2 hrs	24 hrs	Medium bond strength, safe to skin.
TB1324	Red	600	100~150	250~300	1 hr~2 hrs	24 hrs	Medium strength fastening and sealing of screws
TB1374	Red	750	100~150	150~200	1 hr~2 hrs	24 hrs	Effective lubrication, fastening of high tensile strength bolt
TB1327	Red	2500	200~300	200~250	1 hr~2 hrs	24 hrs	High viscosity, medium strength type, fastening and sealing of screw with large clearance
TB1342	Blue	150	50~70	50~70	1 hr~2 hrs	24 hrs	Low bond strength, fastening and
TB1344	Blue	600	50~70	50~70	1 hr~2 hrs	24 hrs	sealing of removable screw

4-2 Effects of Metal Surface Quality to Curing Speed and Bonding Strength

The data of torque and curing speed given in Table of 4-1 refer to uncoated bolt and nut, and these values may vary depending upon the conditions of metal surface. This is attributable to the rate of decomposing peroxide by metal ion (represented by Fe^{2+} in the reaction formulae)

in the initial reaction in the reaction mechanism described in 2-3. While the decomposition rate is supposed to be closely related to the redox potential of metal ion, it is not always confirmed that the faster the setting proceeds, the higher the redox potential is.

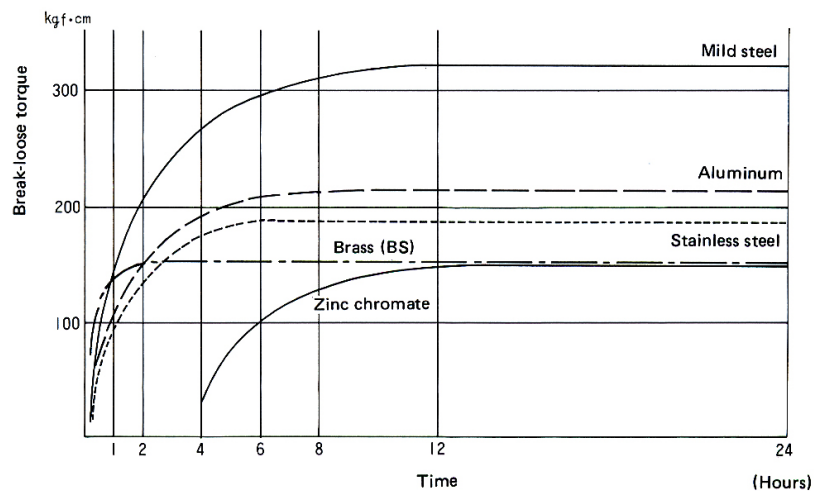
The curing speed and bonding strength of TB-1303 are shown for various surface quality.

Effects of Metal Quality to Curing Speed and Bonding Strength of TB 1303

Bolts used: M10 × P1.5 of normal pitch
Curing conditions: 250°C, 60% RH, kgf·cm

Material \ Time	15 min	30 min	1 hr	2 hrs	4 hrs	6 hrs	8 hrs	12 hrs	24 hrs
Mild steel	20	70	150	200	270	300	280	330	320
Zinc chromate	-	-	-	-	30	100	120	150	150
Black	20	100	180	240	320	300	340	310	310
Stainless steel (SUS)	10	90	110	180	170	190	190	180	190
Galvanized	150	300	350	390	390	400	430	430	440
Aluminum	-	70	100	150	220	200	250	200	220
Nickel-plated	50	150	220	260	280	260	280	280	290
Uni-chrome plating	20	100	200	280	350	370	340	330	350
Brass (BS)	70	140	150	150	150	150	160	150	150

The numeric values refer to rupture torque. Mild steel and black bolts are degreased with 1, 1, 1-trichlor ethane.



4-3 Automatic Torsion Torque Measuring System

1) General

This equipment is to measure the torsion torque automatically, and consists of main unit, display and recorder. The main unit provides two signals: torque measured by a strain gauge type detector, and angle measured by a pulse-type angle detector. The display unit gives digital indication of torque, holds the peak value at starting, and digital reading of torsion torque at a

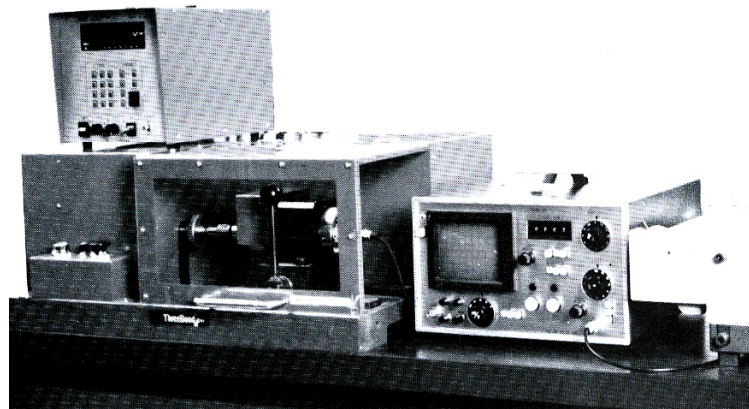
preset angle.

The X-Y recorder continuously records the torque in relation to angle and outputs digital reading on a printer.

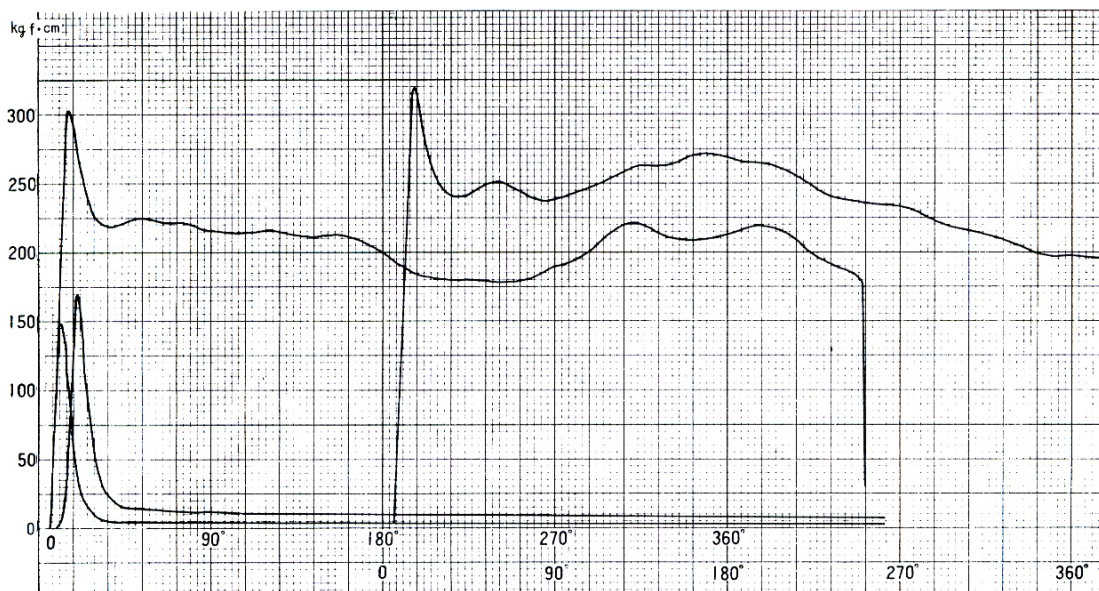
An optional accessory is available to A-D convert fast analogue event by using a transient converter, to store signal in a semiconductor memory, to play back on a furnished oscilloscope and to register on a recorder with readout pulse speed reduced.

This system allows to determine torque of AAS exactly and without involving individual difference.

2) Measuring Equipment and Chart Recording



Automatic torsion torque measuring equipment



Torsion torque (rupture torque and pull-out torque),
Testing method M10×P1.5 mild steel bolt-nut used

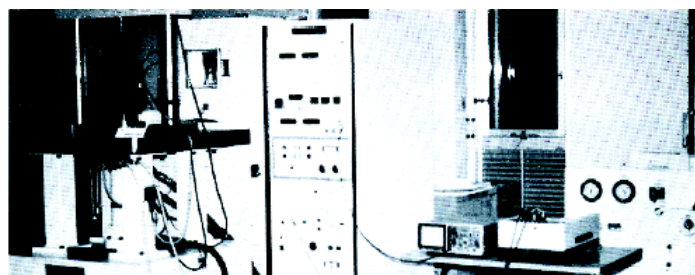
4-4 Locking Function

The screw locking agent is required to have adequate bonding strength, and to be used while grasping the relationship to screw strength by measuring the fastening (axial) force.

Recently, the axial force is measured by hydraulic pressure, supersonic or strain gauge, depending upon screw types. Moreover, the

evaluation of loosening under the dynamic conditions such as vibrations has been advanced. A cross-axial vibration tester (photo) is most frequently used for this purpose, allowing to make comparative tests for various locking mechanisms in a short time. A number of reports have been published by using this machine.

It should be noted that the locking effect of AAS is highly evaluated.



Linear type vibration and stress tester

5. AAS for Fitting

AAS for fitting has been developed for fastening and sealing axial fitting parts. As the fitting involves structural parts, AAS for fitting is required to have better adhesiveness,

chemical resistance, moisture resistance, heat resistance and higher durability under severe conditions than that for screw.

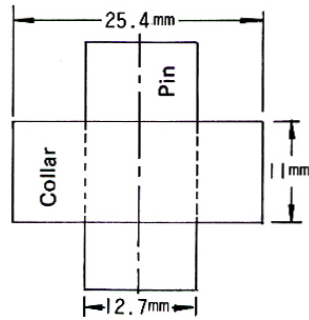
Test data for Three Bond AAS with fitting test pieces are shown below.

5-1 Characteristics of AAS for Fitting

Type		1 3 7 3 B	1 3 7 5 B	1 3 7 7 B	1 3 7 9 B
Color and Appearance		Green, transparent liquid	Green, transparent liquid	Green, transparent liquid	Red, transparent liquid
Viscosity (25°C, cps)		125 ± 25	800 ± 300	2000 ± 500	7000 ± 1000
Shear strength of fitting (kgf/cm ²), steel/steel*1		250~300	230~280	230~280	230~280
Optimum clearance (mm)		0.005~0.01	0.005~0.03	0.01~0.05	0.01~0.05
Maximum clearance (mm)		0.15	0.15	0.3	0.3
Curing speed	Time to usable strength (20~25°C)*2	Within 1 hour	1~2 hours	1~2 hours	1~2 hours
	Time to ultimate strength (20~25°C)	6 hours	12 hours	12 hours	12 hours
Applicable temperature range		-80~150°C	-80~175°C	-80~150°C	-80~150°C
Anticorrosiveness		Excellent	Excellent	Excellent	Excellent
Main features		low viscosity, fast setting, high strength, for general purpose	medium viscosity, high strength, heat resistive	high viscosity, high strength	very high viscosity, high strength
Main applications		fitting in general, particularly for fast setting	fitting in general, particularly for heat resistive use	fitting of medium to large parts	fitting of medium to large parts

*1. With test-pieces illustrated on the next page.

*2. Usable strength refers to 50% of ultimate strength.



Steel pin and collar
 12.7 ϕ x 11t Bonding area 4.39cm²
 Surface finish 6 S
 Clearance 5/100mm
 Compressing rate 1.5mm/min

5-2 Effects of Pretreatment to Bonding Strength

When using AAS, the pretreatment of

bonding surface often comes to matter. The effect of presence of water or oil to bonding strength is tested with TB 1373B. The results are tabulated below.

Test Results

Test-piece Pretreatment Method		Unit (kgf/cm ²)	
		12.7 ϕ steel/steel	6 ϕ BS/SUS
Rinsing with MEK		270	240
Rinsing with 1, 1, 1-trichlor ethane		280	300
Distilled water	A	160	150
	B	290	270
Gear oil	A	150	130
	B	250	250
Cutting oil	A	190	200
	B	250	270

Test-piece
 12.7 ϕ Steel pin and collar
 12.7 ϕ x 11t Bonding area
 Surface finish 6 S
 Clearance 5/100mm
 Compressing rate 1.5mm/min

6 ϕ pin SUS color BS
 6 ϕ x 5t Bonding area 0.942cm²
 Surface finish 6 S
 Clearance 1/100mm
 Compressing rate 1.5mm/min

Method of pretreatment
 A: Pin dipped in water and oil, taken out, and left on rag for 3 min.
 B: Pin dipped in water and oil, taken out and wiped thoroughly with rag.

Applying and setting conditions.
 After the pretreatment, test-pieces are coated with TB 1373B so as to cover the whole periphery, assembled, set at 25°C for 24 hours and put to test.

On the basis of these tests, it may be concluded that wiping with rag provide bonding strength as much as 80% of that with test-piece degraded by solvent. It should be noted, however, that fluctuation in strength and setting

failure may occur in some grade of AAS, oil type or nature of wetting fluid. It goes without saying that the best way is to wash with solvent.

5-3 Fatigue Test

The results of fatigue test which provide important clue for durability will be described in reference to the test with TB1375B.

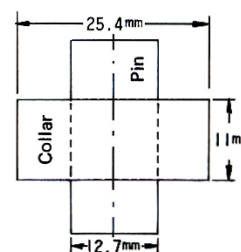
1) Test Conditions

A test-piece as illustrated at the right is coated with TB1375B and fitting was allowed to set at the room temperature.

Material pin S45C (hardened)
 collar S45C (uncoated)

Clearance 5/100 mm

Bonding area 4.39 cm²



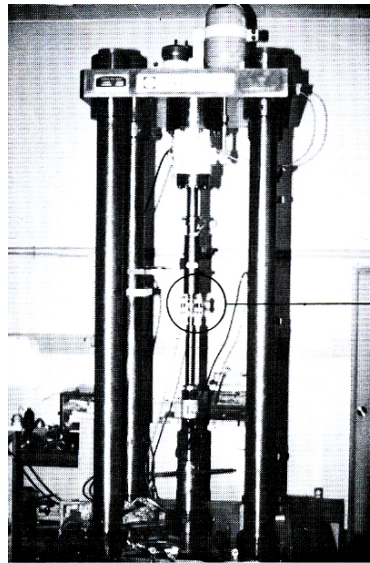
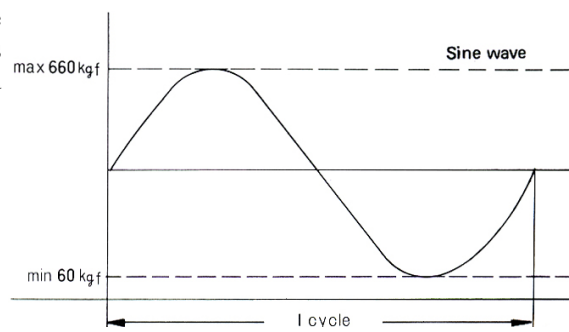
2) Load Conditions

With a load factor about 0.1, the tensile load is constantly kept applied. The test was conducted at 4 points with regard to load conditions.

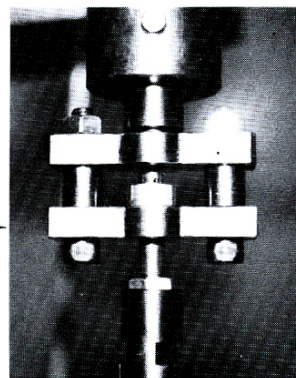
- 60 – 660 kgf
- 50 – 530 kgf
- 40 – 440 kgf
- 35 – 350 kgf

3) Repetition Rate 30 Hz

4) Load Example and Waveform

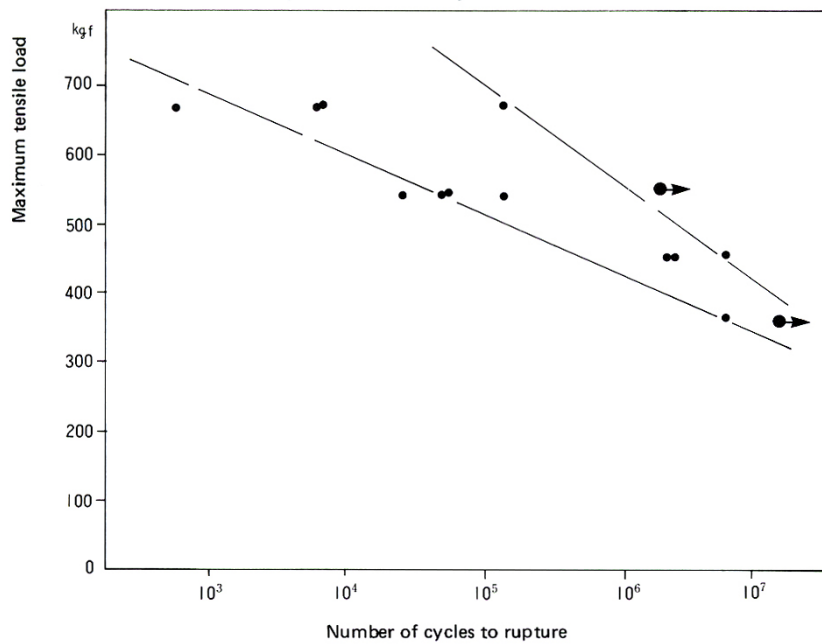


Fatigue test machine



Magnified view of test-piece mounted on the test machine.

Test results Fatigue test with test-piece bonded with TB1375B

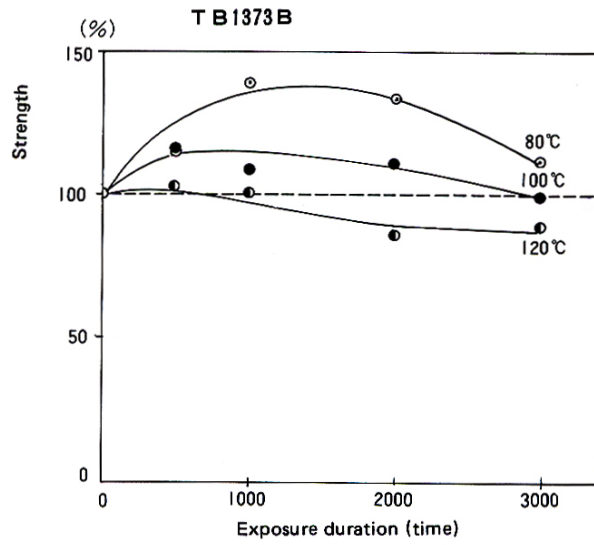


It is said that fitting seldom fails if durability is more than 10⁷ cycles.

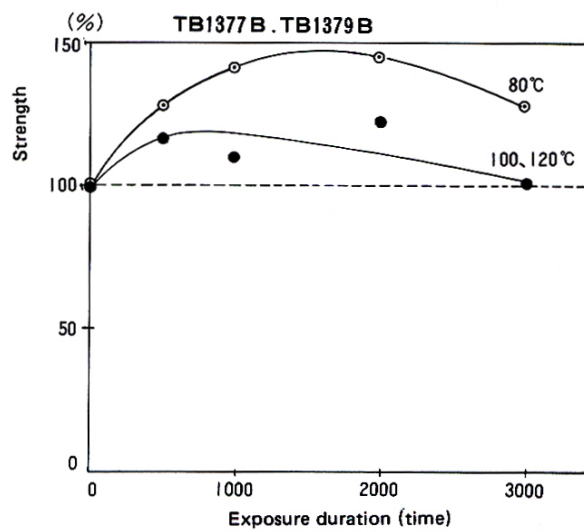
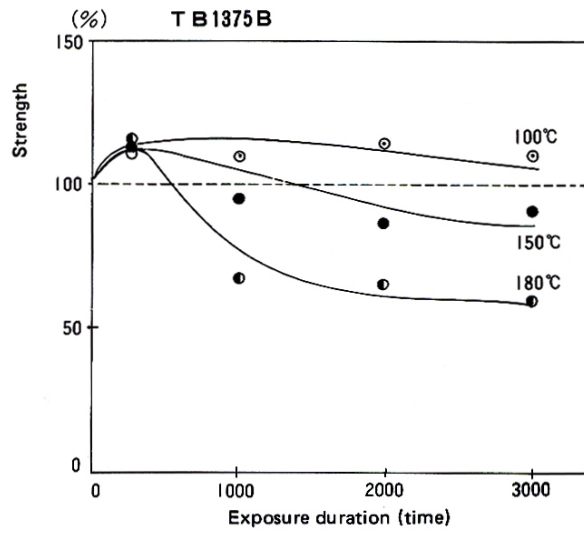
5-4 Thermal Aging Test

Bonded test-pieces are exposed to various temperatures for specified time intervals, and

changes in strength with respect to the initial level are measured. TB1375B gives the best heat resistivity.

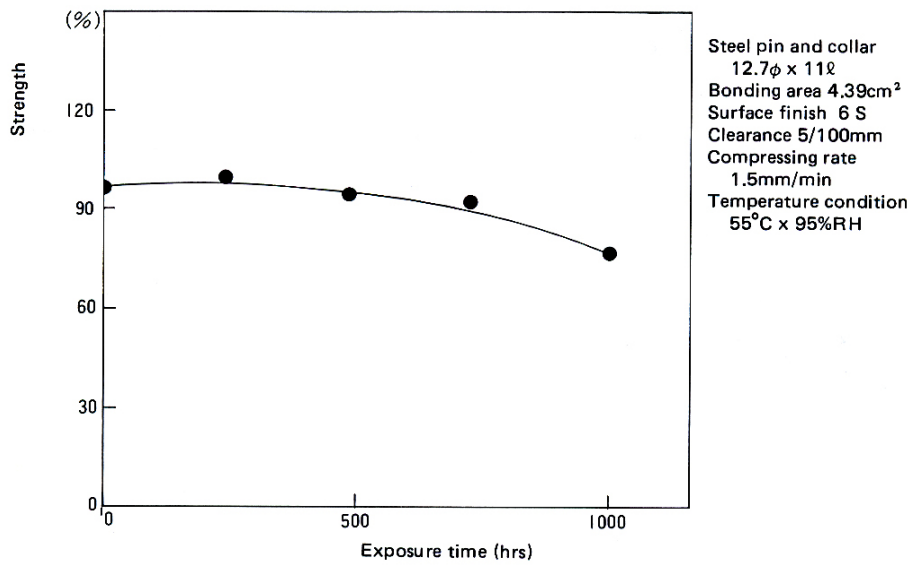


Test-piece
6φ x 15ℓ
Clearance
1/100mm
Setting condition
at room temperature,
for 24 hours
Compressing rate
10mm/min



5-5 Moisture Test

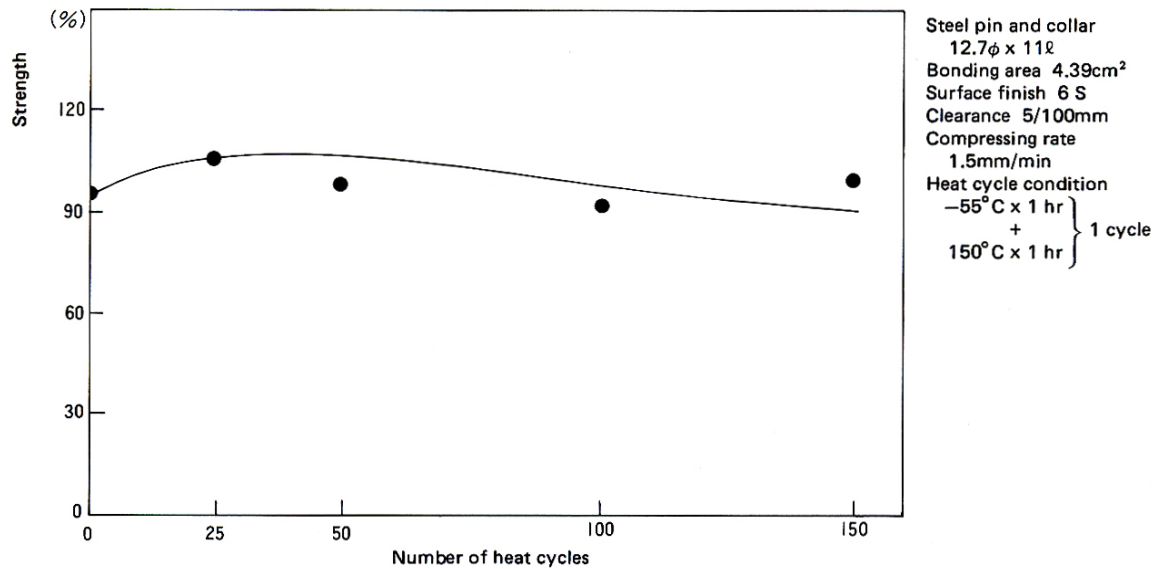
The results of moisture test which is important to electric components as obtained with TB1375B are shown below.



As shown in the above, TB1375B presented the best moisture resistance.

5-6 Heat Cycle Test

Test-pieces conded with TB1375B are subjected to heat cycle, and the strength is followed.



TB1375B presented stable characteristics in the heat cycle test.

6. Special Purpose AAS

Three Bond AAS for special purposes other than screw and fitting are presented below.

6-1 AAS for Back-Impregnation and Nest-Embedding

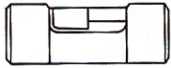
Item \ Grade		T B 1 3 6 1 B	T B 1 3 7 0 B
Color		Green	Blue
Viscosity CPS (25°C)		15 ± 5	125 ± 20
Specific gravity (25°C)		1.080 ± 0.005	1.070±0.005
Bolt fastening strength with mild steel M10 bolt-nut	Break-loose	70~100	180~240
	Prevailing	200~350	60~80
Time to usable strength		15 min~1 hr	30 min~1 hr
Time to ultimate strength		12~24 hrs	24 hrs
Main features		Fast setting, good impregnation	Fast setting, good spray applicability (little splash), good impregnation good weather resistance
Main applications		Back-impregnation for screw, rest-embedding of pin hole	Tire wheel rim, sealing of weld

6-2 AAS for WELCH Plug

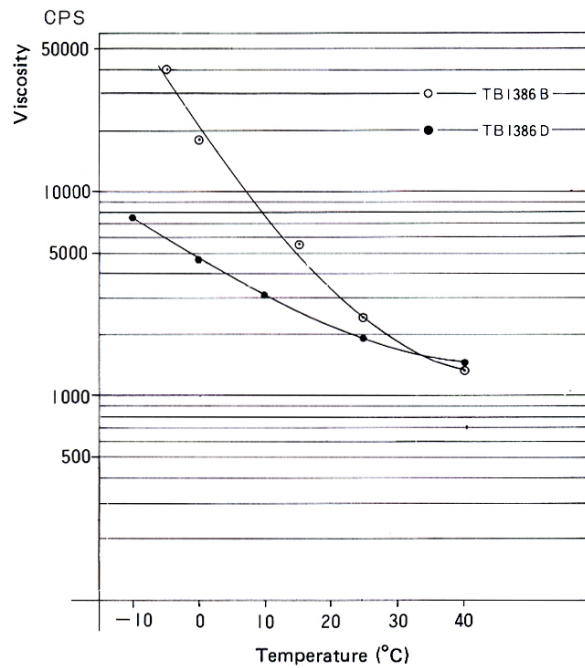
This group includes sealant for sand hole plugging of engine block and head, TB1386D which is the newest grade of TB1386 series.

TB1386D is an improved version of TB1386 series, with better sealing property by providing lubrication property while suppressing viscosity change due to temperature.

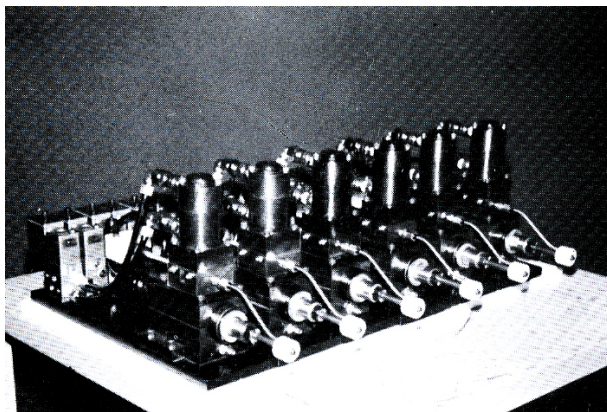
Characteristics of TB1386D

Item	Characteristics	Remarks
Appearance	Red	BL type 30 rpm M10 × P1.5 mild steel bolt-nut
Viscosity	1800 ± 500	
Thixotropic ratio	3 or greater	
Break-loose torque (kgf·cm)	100 ~ 150	
Prevailing torque (kgf·cm)	100 ~ 200	
Time to usable strength	4 ~ 6 hrs	
Time to ultimate strength	24 ~ 48 hrs	
Sealing strength (kgf·cm ²)		with plug 40φ
Initial value	60 or more	within 24 min after plug hammered
Steady state value	80 or more	24 hours after plug hammered
Thermal aging	80 or more	150°C × 120 hrs
Resistance to antifreezing mixture	80 or more	RT × 120 hrs + 100°C × 240 hrs
Resistance to engine oil	80 or more	RT × 120 hrs + 120°C × 240 hrs
Applicable temperature	-50 ~ 150°C lubricating,	illustration of plug hammered 
Other features	small change in viscosity due to temperature	
		tolerance 0.28 ~ 0.40

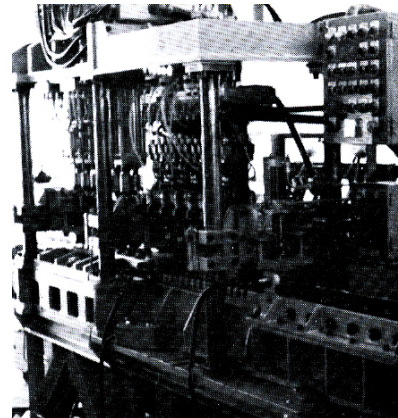
Changes in viscosity in relation to temperatures are shown graphically.



System operation with applicator



6-head rotor type applicator



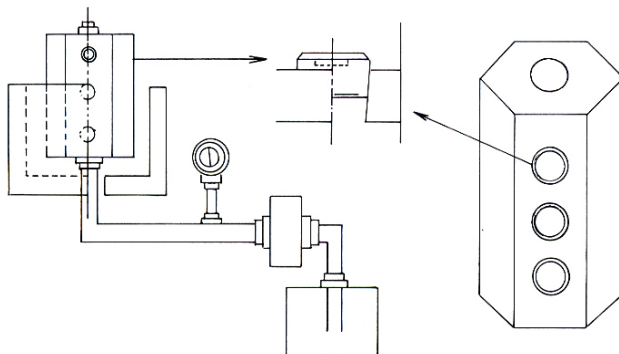
Application system incorporated

7. AAS for Sealing

7-1 Sealing Test with Taper Plug

Sealing with TB1110B using an equipment

shown below and under conditions given below proved that AAS had adequate sealing function.



Test conditions

Plug	SS41 3/4 inch plug
Setting conditions.....	at room temperature (25°C) for 24 hrs
Pressurizing medium.....	turbine oil #1
Pressuring rate.....	5 kgf/cm ² , at every 5 min
Number of test pieces.....	10
Test temperature.....	room temperature, pressurized at 150°C, pressurized
Plug fastening.....	200 kgf-cm 600 kgf-cm

Test Data

Product	Applied Pressure (kgf/cm)	Fastening torque	200 kgf-cm		600 kgf-cm		Evaluation
			Temperature	RT	150°C	RT	
TB1110 B	200	Number of Leaking Specimens	0/10	*1 4/10	0/10	*2 1/10	*1 Leakage occurred in 4 specimens at 115 kgf/cm ² *2 Leakage occurred in 1 specimen at 185 kgf/cm ²
Teflon Tape	200	Number of Leaking Specimens	0/5	*3 5/5	*4 1/5	*5 1/5	*3 Leakage occurred in 5 specimens at 110 kgf/cm ² *4 Leakage occurred in 1 specimen at 115 kgf/cm ² *5 Leakage occurred in 1 specimen at 180 kgf/cm ²

The test with P/T plug proved that the sealing property of TB1110B is better than or equal to that of teflon tape. For the application requiring heat resistance, TB1132 resistive up to 180°C is available. In the practical application, TB1110B is used not only for taper plug but also

for sealing threaded pipes.

Moreover, there are sealants for flange, which are described in "Heat-resisting mechanism of reactive liquid gasket" in the Technical News Nos. 1 and 2.

8. Examples of Main Application of AAS

The application of AAS in various industrial fields are listed below.

Field	Application	Field	Application
Automobile	Engine block, Cylinder head sand hole plug, Ring gear mounting bolt, Air cleaner center bolt, Tire wheel weld seal, Crank shaft balancer screw, Transmission gear, Injection pump mounting screws, Cylinder block baffle plate screw, Fuel pump union, Crank case mounting bolts, Carburettor stud bolt, Thermostat, coulking seal, Steering joint set bolt	Hair Dryer	Motor screw
		Communication Equipment	Rod antenna screw
		Microphone	Pick-up screw
		Magnet Relay	Screws, Pipe joint
		Press Machine	Stud bolt, Pipe joint, Bering fitting
Agricultural machine	Tractor gear case stud bolt, Tractor rear axle housing, Rise planter, Planting can, Lid set bolt, Rice planter mission bolt	Pump	High pressure oil pump screws, Fermentation pump pulley
		Machine tool	Shaft fitting, Bush fitting, Valve control bolt, Base-head fitting
Construction Vehicle		Crane	Lever screw, Other screws
Bulldozer	Armor bolts, Engine bolts, Bearing fitting, Wheel plug screw	Printing Machine	Screws, Sleeve filling sealing
Shoval loader	Armor bolts, Engine bolts, Bearing fitting, Wheel plug screw, Bonnet side cover set bolt	Cutting Machine	Screws
		Crusher	Stud bolt, Bush metal fitting
		Industrial Sewing Machine	Two-needle fastening part, Bed fit face
Railway vehicle	Window frame screws, Pipe joint, Bearing fitting, Chassis bottom equipment set bolt, Inside equipment set screws, Generator-motor clearance embedding	Bowling Machine	Seizer deck, Roller bracket, Take lift shaft, Pin-setter bolt
		Oil Cooler	Stud bolts
		Dust Collector	Screws
Ship	Turbine bolt, Union nipple, Piston stud bolt, Shaft pin fitting, Head set bolt, Pipe joint	Bread Steamer	Door screws
		Cash Register	Screws
		Electric Appliance	
Motor	Stepping motor, Shaft fixing, Axial flow Fan motor case/yoke fixing, Coreless motor shaft fixing	Wood-working Machine	Screws
		Transmission	Sliding pulley
VTR	Head bearing fitting fixing	Centrifugal Clutch	Blade fixing
Generator	Shaft-pulley fitting, Runner bolt, Line hardware bolt	Valve	Seat ring fitting
		Electrical Type-writer	Bearing fitting, Screws
Electric Washer	Hydroextractor shaft set screw	Water Supply Hardware	Screws
Tape Recorder	Shaft-pulley fitting and screws		
Vacuum Cleaner	Bearing fitting		

Conclusion

Since AAS is special adhesive and sealant, there is little literature on it. We are happy if this report would be helpful to your business or research. In near future, it is expected that the following problems would come to matters.

1. Improvement of heat resistance.
2. Curing property of thick film
3. Curing property on oily surface
4. Effect to plastics
5. High bonding strength, softening, compositeness, etc.

We are going to improve the technical level so as to meet users' requirements.

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