

## Study on Heat Resistance Anaerobic Adhesive Which Expands When Post Cured

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### ABSTRACT

In this paper, N,N-m-phenyenedimaleimide (m-PDM), Polyamide Resin (PI) and Metallic dimethacrylate etc. influencing the heat resistance of anaerobic adhesive has been studied, an anaerobic adhesive composition capable of post-cure expansion and 230°C temperature resistance comprising a multifunctional methacrylate and m-PDM capable of effectuating expansion upon post-cure has been developed. A homogeneous mixture of a monomer and m-PDM are subjected to a first cure heat stage (Room Temperature) wherein a rigid partially-cured plastic is formed and a post-cure heat stage (150°C) to effectuate permanent expansion of cured adhesive composition.

### INTRODUCTION

In this paper, N,N-m-phenyenedimaleimide (m-PDM), Polyamide Resin (PI) and Metallic dimethacrylate and epoxy novolac acrylate influencing the heat resistance of anaerobic adhesive has been studied, an anaerobic adhesive composition capable of post-cure expansion and 230°C temperature resistance comprising a multifunctional methacrylate and m-PDM capable of effectuating expansion upon post-cure has been developed. A homogeneous mixture of a monomer and m-PDM are subjected to a first cure heat stage (Room Temperature) wherein a rigid partially-cured plastic is formed and a post-cure heat stage (150°C) to effectuate permanent expansion of cured adhesive composition.

### EXPERIMENT

The composition of the heat resistance anaerobic adhesive

Based prescription	(Total 100 by weight)
Ethoxylated bisphenole A dimethacrylate	70~80
Hydroxypropyl methacrylate	1~5
Heat resistant agent	10~20
Saccharin	0.1~1
Maleic acid	0.1~1
Additive(to be specified)	0.5~1.5
Stabilizer	0.5~1.5
<i>Cumene hydroperoxide</i>	2~3
Dye	0.1~0.5
Silica,amorphous, fumed, crystalline-free	0.5~2

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The property results of the anaerobic adhesive which added deferent heat-resistant agent after 24 h cure at room temperature

(1) Sample making

According to JB/T7311-94 standard, the compressive sheer strength sample as Figure 1.

(2) The test results list in Table 1

The property results of the anaerobic adhesive which added deferent heat-resistant agent after 24 h cure at room temperature and 1 h post cure at 150°C

(1) Sample making

According to JB/T7311-94 standard, the compressive sheer strength sample as Figure 1.

(2) The test results list in Table 2

Expend rate of the anaerobic adhesive which added deferent heat-resistant agent after 1 h post cure at 150°C

(1) Sample making

The sample is  $\phi 20$  mm, 30 mm deep hole

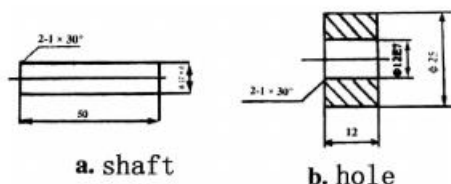


Figure 1. The sample of compressive shear strength.

in circular cylinder which made of still, anaerobic adhesive pour in it at 20 mm deep, then put it into oven , keep it one hour at 150°C, after that test the volume change.

(2) The results list in Table 2

## ANALYSIS OF THE EXPERIMENT RESULTS

From Table 1 we can find that adding, N,N-m-phenyenedimaleimide(m-PDM), Polyamide Resin(PI), Metallic dimethacrylate and Epoxy novolac acrylate can improve the heat resistance property of anaerobic adhesive , the effects of the 4 materials are very like when the adhesive cured at room temperature (as Table 1), but it is very deference after the adhesive post cured 1h at 150°C. Comparing Table 1 with Table 2, the compress sheer strength of the adhesives which added Metallic dimethacrylate and epoxy novolac acrylate is not changed, but the compress sheer strength of the adhesives which added N,N-m-phenyenedimaleimide(m-PDM), Polyamide Resin(PI) is increased , especially at 200°C and 230°C. Why so? Now lets us analysis the IR picture of the adhesive which added m-PDM when cured at room temperature and 150°C.

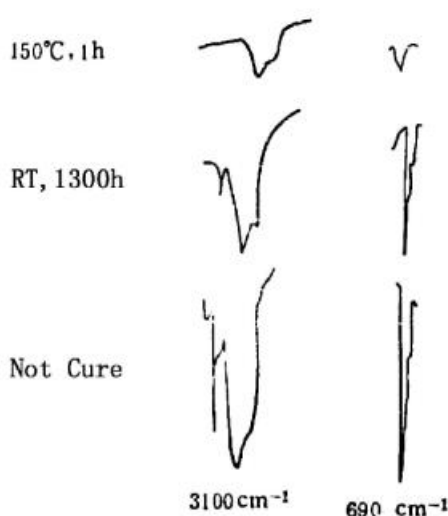
Figure 2 shows the changes of m-PDM feature peak at  $3100\text{ cm}^{-1}$  and  $690\text{ cm}^{-1}$  of the adhesive which added m-PDM when not

Table 1. Compressive Sheer Strength of the Anaerobic Adhesive which Added Deferent Heat- resistant Agent (After 24 h cure at room temperature)

Heat resistant agent	Properties	Stability (Gel time at 82°Cmin.)	Compressive sheer strength (M Pa)			
			R.T	150°C	200°C	230°C
No heat resistant age		120	21.6	13.2	0	0
Epoxy novolac acrylate		60	20.8	18.2	3.1	0
Sare modified metallic dimethacrylate		30	22.1	21.8	2.9	0
Polyamide resin(PI)		60	15.3	10.2	2.5	0
N,N-m-phenyenedimaleimide(m-PDM)		120	16.9	11.2	2.9	0

**Table 2. Compressive Sheer Strength of the Anaerobic Adhesive which Added Deferent Heat-resistance Agent (After 24 h cure at room temperature and 1 h post cure at 150°C)**

Heat resistant agent	Properties	Expend rate (%)	Compressive sheer strength (M Pa)			
			R.T	150°C	200°C	230°C
No heat resistant agent		-0.01	21.9	13.5	0	0
Epoxy novolac acrylate		-0.02	21.2	22.5	2.2	0
Saret modified metallic dimethacrylate		-0.02	22.1	21.1	1.9	0
Polyamide resin(PI)		0.09	23.5	21.0	16.8	4.9
N,N-m-phenyenedimaleimide(m-PDM)		0.11	24.6	24.3	23.5	13.2

**Figure 2. IR of m-PDIM modifying anaerobic adhesive at different temperature.**

cure, after 1300 h cure at room temperature and after 1 h cure at 150°C. The peaks are same between not cure and 1300 h cure at room temperature, but the peak is changed after 1 h cure at 150°C, so we know that from Figure 2, m-PDM is not take part in chemical reaction at room temperature, it is just at above 150°C high temperature, m-PDM can crosslink with other materials,

improving the heat resistance property of the adhesive, another, the adhesive which added m-PDM is expend after 1 h post cure at 150°C, so the dowel joint compress sheer strength is increased at high temperature.

## THE PROPERTISE OF THE HEAT RESISTANT ANAEROBIC ADHESIVE WE DEVELOPED

An anaerobic adhesive composition capable of post-cure expansion and 230°C temperature resistance comprising a multifunctional methacrylate and m-PDM capable of effectuating expansion upon post-cure has been developed, its properties list in Table 3.

## CONCLUSIONS

(1) Adding, N,N-m-phenyenedimaleimide (m-PDM), Polyamide Resin (PI) can improve the heat resistance property of anaerobic adhesive.

**Table 3 The Properties of Teat Resistance Anaerobi Adhesive which Added m-PDM**

Color	Density (g/cm <sup>3</sup> )	Viscosity (mPa.S)	Fixturing time (min)	Stability (Gel time at 82°C, min.	Compressive sheer strength (R.T, MPa)	Compressive sheer strength (230°C, MPa)
Green	1.05	7000	15~30	≥ 2	25.6	15.2

(2) m-PDM is not take part in chemical reaction at room temperature, it is just at above 150°C high temperature, m-PDM can crosslink with other materials, improving the heat resistance property of the adhesives.

(3) The anaerobic adhesive which added m-PDM is expend after 1 h cure at 150°C, so the dowel joint compress sheer strength is increased at high temperature.

## REFERENCES

1. Lu Qiting, "Fast cure adhesives", Science Press of China (1992).
2. Zhai Haichao, "Adhesives prescriptions and ways to produce" (2000).
3. Japan patent, JP04323282 (1992,11,12).
4. World patent, WO9403504 (1994,2,17).