Stress Failure of Airborne Optical Assembly Resulting from Rigid, High Modulus Epoxy Adhesive and Quality Improvements

Jun Xiao,[1,2] Hong-su Li,[3] Xin Lu,[2] Jian-qi Meng[2]

[1]*Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, People's Republic of China*
[2]*014 Center of the First Aviation Group Corporation of China, Luoyang 471009, People's Republic of China*
[3]*College of Material Science and Engineering, Northwestern Polytechnic University, Xi'an 710072, People's Republic of China*

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ABSTRACT: Up to 40% failure odds on an airborne optical assembly were found during finalizing the design-production phase. It resulted in lens splitting, cracking, and shattering. The combined stress of residual stress originated from solidification shrinkage and deformation stress and from temperature changes that drastically caused the failures. The optical assembly was composed of aluminum shell, rigid epoxy adhesive layer, and glass lens. Mechanism and affecting factors of the failure were investigated on process, operation, and materials. A series of comparative trail experiments were carried out. It was recommended to prevent the failures by redesigning match clearance between duralumin wall and lens, replacing rigid epoxy adhesive with flexible polyurethane adhesive. Via these new measures, all optical assemblies made hereafter succeeded and passed all military environmental tests and inspections, with a zero percent failure odds. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 99: 45–51, 2006

Key words: airborne optic assembly; stress failure; adhesive; process techniques

INTRODUCTION

Optical assembly composed of dome kit and lens kit is crucial to certain airborne searching system. But development and design finalizing of the system were restricted by up to 40% failure rate. Lens splitting, cracking, and shattering were obviously found (see Fig. 1) after Chinese military environment condition tests, especially in high and low temperature, thermal shock and vibration tests. Statistics shows that there was a failure rate of 90% and a serious failure rate of 40% during finalizing of the design trial production. Here, tens of products had delivered and others were followed up for the coming flight and field test for design finalizing. It was urgent to solve the following problems: (1) What were the major causes resulting in high failure rate? (2) Why was the failure rate in design finalizing (D) stage much higher than in first article study (C) stages and experiment prototype study (S) stages? (3) Could products that passed military inspection and accepted be applied in the coming field tests for design finalizing? (4) How to avoid high failure rate and guarantee quality and eligibility for subsequent production?[1] In this article, failure analysis, trial experiments, and some efficient improving measures were discussed.

EXPERIMENTAL

Raw material

Stycast2850FT epoxy adhesive and Catalyst Nos. 9 and 11 (Emerson and Cuming Co.), RTV560 + T12 silicone rubber adhesive and primer SS 4004 (GE Co.), Sy2850 epoxy adhesive/Cat9, Cat11 (Beijing Academy of Aviation Material, China), and polyurethane primer PR420 (PRC-DeSoto Co., USA) were used. Polyurethane elastic adhesive PU-2 was prepared in our lab. KH-560 and other auxiliary materials are made in China.

Sy2850FT is a two-part, electric encapsulanting epoxy adhesive. It is composed of bisphenol A epoxy resin (No. 618) and epoxy living monomer (No. 501). To lower the coefficient of thermal expansion and get a stable size, fillers such as alumina, silica, black dyestuff, and defoamer were added. Sy2850FT can be used with two curing agents Cat 9 (aliphatic amide catalyst, curing at room temperature for 48 h) and Cat 11 (mixed aromatic amide catalyst, curing at 100°C for 4 h). They were similar to Stycast 2850FT, catalysts No. 9 and 11 of epoxy adhesive of Emerson and Cuming Co. products.

Correspondence to: H-s. Li (hlshong@163.com).

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PU-2 is also a two-part polyurethane adhesive. Part A is prepared by reacting poly(tetramethylene glycol) (PTMG) with toluene 2,4-diisocyanate and additives. Part B is prepared by reacting 3,3′-dichloro-4,4′-diamino-diphenylmethane with PTMG. When adhesive is needed, mix part1/part2 (1.9/1, w/w), degas, and cure at room temperature for 24 h and at 80°C for 24 h.

KH-560 is a silane coupling agent. It was prepared with a 0.5% (w/w) alcohol solution as primer to enhance bonding between epoxy adhesive and abstract.

Instruments and equipments

A programming environment condition tester (SE-600–5-5) was employed to carry out hot and damp, high and low temperature tests and temperature shock test. A salt fog tester (YL-40C, China) was adopted for salt fog corrosion test and an electromagnetic vibration equipment (V964LS, England LDS Co.) for vibration test. An all-purpose electric tester (Instron 5581, Instron Co.) was adopted for strength test.

Assembly adhesive and leakage inspection

(1)Adhesives were prepared according to manufacturer's instructions and degassed under vacuum, at 5×10^{-4} Pa for 5 min. (2) Leakage inspection in anhydrous alcohol was done at minus pressure, 0.7 kg/cm^2, to check whether some air bubbles could be seen at the joint.

Environment condition tests

Environment condition tests were carried out according to high temperature test (GJB150.3; (65 ± 2) °C for 48 h), low temperature test (GJB150.4; (−55 ± 2) °C for 48 h) and temperature shock tests (GJB150.5; (60 ± 2) °C for 3 h and (45 ± 2) °C for 3 h; conversion time <5 min, proceeding three circulation). Other environment condition tests include vibrations test (GJB150.16), hot and damp test (GJB150.9), and salt fog test (GJB150.11).

RESULTS AND DISCUSSION

Failure and stress concentration

Figure 1 illustrates the configuration of the optical assembly composed of hard duralumin shell or dome, glass lens, assembly epoxy adhesive layer, and a gasket between the lens and metal wall. Since most of the failures were found during environment condition tests and since thermal expansion coefficients of the glass, metal and epoxy resins are different, some undue factors or stress were initially reckoned as major causes, resulting in failures during high and low temperature, thermal shock and vibration tests or rolling process. However, in D, C, and S stages, technical state and conditions, such as materials, match clearance, adhesive, process, and operators, were similar except rolling, but the failure rate increased. So three aspects were expected to have stress concentration for the failure:[2]

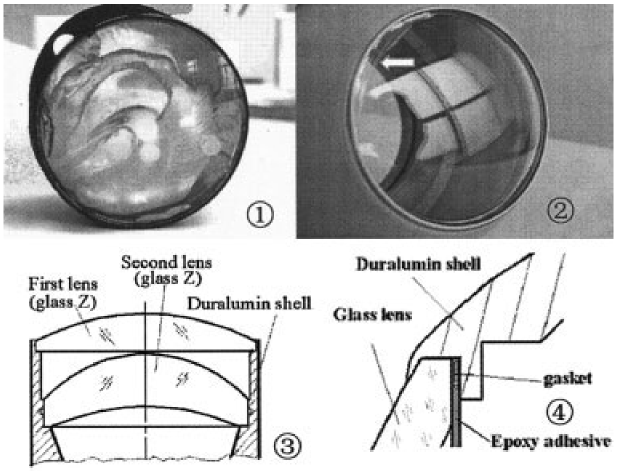

Figure 1 Sketch of failure optic assembly and its structure. ① Failure lens kit, ② Failure dome kit, ③ and ④ Structure of lens.

1. Rolling stress: Undue operation and process might distort the edge of metal dome. It led to prestress on the lens.
2. Thermal stress: Different thermal expansion coefficient of glass, metal, and epoxy adhesive led to different deformations, leading to high stress when temperature changed drastically.
3. Solidification stress: High strength and rigid structure of the epoxy adhesive may result in high stress during solidification (crosslink reaction of epoxy resin), which may slowly slack as time passes and depending on the resin and structure, holes or lacuna increase stress.

Stress analysis

To find out the major causes resulting in failures, the level of thermal stress of the optical assembly was studied. A simplified model, composing of three parts, represents the optical assembly employed. The dome was simplified as a thick round tube, with the optical lens as a column (see Fig. 2). A second lens is taken as an example, where r is the radius from the center of a circle, a is the inner radius of model, and b is the external radius. We assume that the thick round tube subjects even internal pressure P_a and external pressure P_b. Match clearance increases or decreases at high and low temperature for quite different thermal expansion or contrast for duralumin shell and optical glass lens. Since epoxy adhesive layer is very thin and

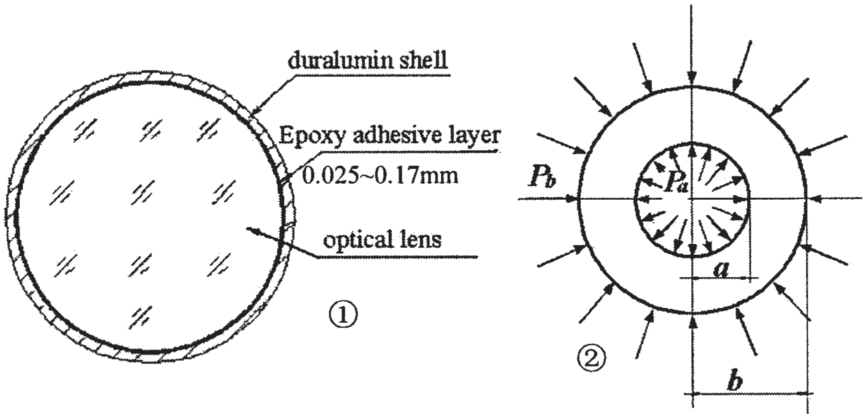

Figure 2 Simplified model for thermostress calculation. ① Cross section of simplified model, ② Round thick tube with even stress.

hard, inducing interactive stress, its elastic deformation could not match the changes of clearance. Consequently, we could assume that there were equal stresses distributing on duralumin shell wall and inside and outside very thin adhesive layer. Therefore, a typical resolution for stress analysis on plane in elasticity mechanics could be cited—a model of a round thick tube enduring even pressure, as shown in ② of Figure 2: By making use of boundary and single displacement conditions, radial stress σ_r and circumferential stress σ_θ could be gained in eqs. (1) and (2) as follows:[3]

$$\sigma_r = \frac{P_a a^2 - P_b b^2}{b^2 - a^2} + \frac{a^2 b^2 (P_b - P_a)}{b^2 - a^2} \frac{1}{r^2} \tag{1}$$

$$\sigma_\theta = \frac{P_a a^2 - P_b b^2}{b^2 - a^2} - \frac{a^2 b^2 (P_b - P_a)}{b^2 - a^2} \frac{1}{r^2} \tag{2}$$

To determine radial stress, extreme temperature changes ($\Delta T = 80°\text{C}$) were calculated with the thick tube model—for the shell, $P_b = 0$, $a = 24.5$ mm, and $b = 26.7$ mm, and for lens as a round tube with $P_a = 0$, $a = 0$, and $b = 24.5$ mm. Then, radial displacement u may be expressed by eq. (3) as follows:

$$u = \frac{1}{E(b^2 - a^2)}\left[(P_a a^2 - P_b b^2)(1 - \mu) r - a^2 b^2 (P_a - P_b)(1 + \mu) \frac{1}{r} \right] \tag{3}$$

where E is the material's elastic modulus, and μ is the Poisson's rate. For Z glass, $E_B = 54.7$ GPa and $\mu_B = 0.216$; For duralumin shell wall, $E_{\text{Al}} = 71$ GPa and $\mu_{\text{Al}} = 0.33$. Elastic modulus E of epoxy adhesive was not available, with only a rough range of 7–15 GPa. Taking an average value $E = 10$ GPa and $\mu = -1$, to the most disadvantageous circumstance, total displacement of three parts by radial stress, P, is just the same as redundant shrinkage. Establishing equations according to the conditions, the stress P_b on lens out-surface is about 5–6 MPa, the same as circumferential stress.

Simplifying the features of original structure and size, it is tantamount to decrease the effects of exterior deformations, and so the actual stress should be greater and not be neglected. The calculations show that deformation of such a thin, hard, and high elastic modulus of epoxy adhesive layer is unable to release high thermal stress. The second lens is taken as an example, which endured major radial shear stress and is easily broken, as shown in Figure 3, and the critical shear strength of the glass lens is about 5–6 MPa.

Stress failure tests

Influence factors

To better study the failure, nine optical assemblies, five sets bonded with epoxy adhesive Sy2850FT + Cat 9 (aliphatic amide catalyst) and Cat 11 (mixed aromatic amide catalyst), and four sets bonded with Stycast2850FT + Cat 9 epoxy adhesive, went through trail tests, with specifications after preleakage inspection. Flaws were found with leakage on two unrolled domes after high temperature test. Flaws and leakage

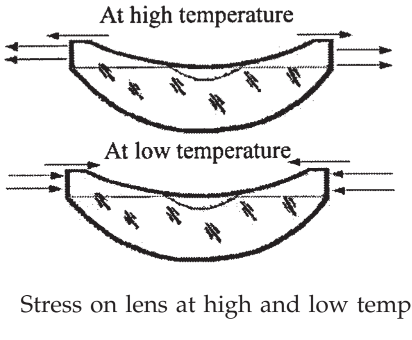

Figure 3 Stress on lens at high and low temperature.

TABLE I
Match Clearance at Room Temperature, High, and Low Temperature

Temperature	Room temperature	High temperature	Low temperature
Tolerance			
Shell	0.10	0.11	0.05
First lens	0.05	0.05	0.01
Second lens	0.035	0.065	
Third lens	0.085	0.098	0.06

were also found on other two rolled domes and four lens kits after low temperature test made a high failure rate up to 88%. These results hinted us that some inherence factors worked. So all influence factors relating to personnel, machine, materials, method, and environment were analyzed:

1. Match clearance: Enduring compress and tensile stress, epoxy adhesive layer might not meet deformations of Al parts expanding and shrinking while temperature changes drastically.
2. Rolling process: It had been validated by tests to have no distinct influence.
3. Optical materials: Materials from different manufactures at D, C, and S design stages might have different strength and properties. It needs to be tested.
4. Personnel: Need to be investigated through tests, supervision.
5. Thermal expansion or shrink: Different expansions or shrink, uneven temperature distribution or fast elevation of temperature in an oven might result in stress on optical parts.
6. Designed and actual clearance: Actual match clearance was usually less than the designed guideline for internal diameter.
7. Different deposited time: High rigid epoxy results in high stress during solidification, and needs longer time to release.(Sy2850FT $\nless$7 days in other similar aviation applications).
8. Bonding: Bonding area and primer KH560 were tested, but no difference was found.
9. Epoxy adhesive: Domestic adhesive might be coarse, with high stress, and cured fast at initial solidification period than imported one. However, there were failures with imported epoxy adhesive. Even curing with aliphatic amide catalyst Cat 9 (cured at room temperature for 48 h), though was better than Cat 11 (cured at 100°C for 4 h) could not avoid failure arises. A study shows that the glass transition point (T_g) and elastic modulus (E) of the adhesive were higher than was stress.

Match clearance changes

Match clearance of each part had been measured actually at different temperatures (see Table I). It shows that match clearance between glass lens and duralumin parts changed greatly at high and low temperatures, which tally with forecast analysis.

Thermal expansion or shrinkage

Coefficients of thermal expansion of glass lens, duralumin shell, and epoxy adhesive layer are shown in Table II. Being very hard for both optical glass and epoxy adhesive, their deformation could be neglected. There were stresses in radial and axial directions. Adhesive layer endured compress and tensile stress from connected parts by deformation and gave limited yield. Clearance increased at high temperature, bringing tensile and shear stress on optical lens, whereas shrinks at low temperature, bringing compress stress and shear stress. Radial epoxy adhesive layer endures thermodeformation stress and gives a limited yield for its hardness, passing shear stress through bonded interface to connected optical lens. Stress in axial direction might be neglected for nonrestricted displacement. To apperceive the magnitude of adhesive layer changes, changes in thickness of an epoxy plate (Sy2850FT, 6 mm in thickness), pretreated at 65°C for 7 h and cooled to room temperature, was actually measured. It was just 0.033 mm only. Hence, the change of adhesive layer for optical assembly (thickness max < 0.2 mm) is ≤ 0.001 mm. At this level it could be neglected compared to Al parts. Therefore, high strength adhesive layer suffers and transfers great tensile and compress stress due on the lens.

Strength of optical kits

Tensile, compression, and tripoint bend strength of two species of glass (K, Z) had been determined (Fig.

TABLE II
Coefficients of Thermal Expansion[a] of Glass, Al Shell, and Epoxy Adhesive Layer

Material		Glass K	Glass Z	Duralumin	Sycast 2850FT
Coefficient of thermal expansion, $\times 10^6$	20~100°C	7.6	8.2	22.7	35.5
	−60~0°C	7.2	7.9	21.4	31.0
Method			ASTM-D-3386		

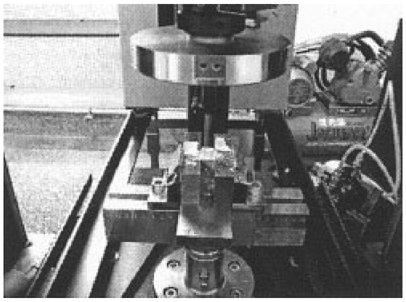

Figure 4 Strength tests on optical samples.

4). No prominent difference in compression strength of lens from different manufacturers was found. However, shear strength of glass, especially for duralumin–glass (lens) joint (Al/stycast2850 FT/glass K), was very low, 2.9/2.5–3.2 MPa only, and shear strength of Al/stycast2850 FT/glass Z was 2.7/2.5–2.9 MPa, even less than the calculated critical value of 5–6 MPa, which corresponds to analysis of a simplified model.

Thermal shock

Eight sets of optical parts were assembled with epoxy adhesive Stycast 2850FT/Cat 9 under strict preparing and degassing ,according to specifications. They had deposited it for 7 days before environment tests. Five unrolled lens kits and five domes were accidentally taken out from an oven heated at 70°C for 20 min, during high temperature storage test. Splitting, cracking, and shattering were obviously found on four lens kits and all domes. Two rolled domes passed leakage inspection, and 4 of 5 lens kits had been found with splitting, cracking, and shattering after storage test at 65°C. Two of seven kits had been found as failures after low temperature (−55°C) storage test. No failure was found in last thermal shock test (−45°C, 2 h). These results indicated differences between optical materials, operators, match clearance, foreign or domestic adhesive, rolling, and bonding style that were not responsible for high failures.

It is shown that neither thermostress nor residue stress of the adhesive could result in failures, respectively. Seven days was not enough for high strength rigid epoxy release solidification stress. Hence, long-time deposition was needed for releasing the stress efficiently after bonding. A survey on relationship of deposition to failure rate shows that the failure rate of 90% was just deposited 7 days, while D stage was 40% deposited for 1 month, and low failure rate at C and S stages deposited after 6 months. This may be the reason why most kits that were produced at C and S stages passed military inspections and their quality was reliable. Stress attenuating kits that passed inspections of D stage are also qualified for the requirements though their stress was not fully released.

Mechanism of the failure

Based on the aforementioned trail tests and analysis, possible failure mechanism should be clearance between optical parts varies as temperature changes. The epoxy layer is very thin rigidly, while thermostress emerges when its elastic strain does not match the changes of clearance. High bonding strength and rigid epoxy adhesive tends to centralize high local stress during solidification (crosslink shrinkage). So the combined centralize stress may cause splitting, cracking, and shattering on optical lens with poor shear and tensile strength.

Improvement measures

Although prolonged deposition time can decrease in failure rate directly in some way, it is not feasible: First, it was difficult to find out exact and proper time. Failure might reappear for insufficient deposition, whereas it greatly prolongs production periods; Second, it only decreases solidification stress and not the combined stress; third, it is not fit for large and multibatch production for poor eligible rate. Hence, two improvement measures were implemented:[4] (a) Replace materials of the shell, dome, and adhesive with closest thermal expansion coefficient to lens; (b) Modify Sy2850FT epoxy adhesive by copolymerization, blending, interpenetrating , and synthesizing with consistent liquid rubber, such as polysulfide, terminal-carboxyl group liquid rubber, hydro-terminal liquid rubber, etc. This would greatly lower its glass transition point (T_g) and elastic modulus (E); (c) Apply new assembly adhesive with excellent elasticity, less solidification stress, and adjust minimal match clearance to an appropriate level. It will greatly slack the stress and environment influence.

It is no doubt that the aforementioned measures would effectively decrease the stress of optical kits and eliminate the failures. But the third measure is more favorable.

How to select assembly adhesive

Principles to select assembly adhesive are as follows:

(1) Being successfully applied on other aviation systems with high quality, constant and abundant accommodation; (2) High flexibility and good strength to bond the joints; (3) less solidification stress; (4) excellent high and low temperature performance and long aging life.

According to earlier principles, silicone rubber RTV560 and polyurethane PU-2 were chosen for improving trail production. In addition, polyurethane adhesive possesses better impact and peeling strength than does epoxy adhesive (See data in Table III).

TABLE III
Performance of Assembly Adhesives

Performance	Silicon rubber RTV 560	Polyurethane PU-2	Epoxy stycast2850FT	Epoxy sy2850FT
Density (kg/m^3)	1.42	1.05–1.20	2.35–2.45	2.35–2.45
Hardness	≥55 S A	≥91 S A	≥88 S D	≥90 S D
Elongation (%)	≥120	≥360	-	-
Tear strength (kN/m)	5.5	80.7	-	-
Working temperature (°C)	−115 to 260	−70 to 120	−40 to 130	<160
Linear shrinkage (%)	0.2–0.6	-	0.002	0.02
Coefficient of expansion(/°C)	2.0×10^{-4}	1.8×10^{-5}	35×10^{-6}	55×10^{-6}
Primer coating	LZ-2, SS 4004	PL-5, PR-420	no	no
A1-A1 shear strength (MPa)	5.2	16	25	25

Estimated joint strength and tests

Strength for dome kit

The greatest pressure (P) for dome to bear could be calculated, according to the maximal pressure of 0.07 MPa; $P = 17.35$ (kg). The minimum round bond area S1 is 3.517 cm^2 and the minimum bonding area S2 on domes is 3.517 cm^2. Then, the greatest pressure around lens was only 0.486 MPa, when its bottom was not bonded. Only 0.25 MPa of shear and tensile strength was needed if bonded. In addition, lens of dome is positioned and held after rolling process. Therefore, bond strength for dome kit with new adhesive does meet the requirements.

Strength for lens kit

Lens kit does not bear positive or negative pressure but mainly axial impact. The maximal shear stress for lens' first layer of adhesive is about 0.01 MPa, under the maximal impact effect in six accelerated gravity. Test with Instron 5581 all-purpose electric machine shows that shear strength of adhesive was about 8 MPa, which fully met the strength requirements of optical kits.

To eliminate thermostress, minimum match clearance, ∢0.07 mm, must be guaranteed while maximal match clearance unchanged to ensure coaxiality. Technical states of match clearance before and after improvements were shown in Table IV:

TABLE IV
Technical States of Match Clearance Before and After Improvements

Parts	Match clearance	
	Before improvement	After improvement
Dome	0.03–0.178	0.06–0.178
First lens	0.03–0.15	0.06–0.150
Second lens	0.025–0.126	0.06–0.139
Third lens	0.025–0.126	0.05–0.129

IMPROVING MEASURES AND EFFECTS

Test project

Based upon mechanism analysis, minimum match clearance ∢0.07 mm was redesigned while maximal clearance unchanged to ensure adhesive layer at appropriate thickness to release deformation stress, as well as guarantee optical coaxial at the same time. Furthermore, bonding optical kits, 10 domes and 10 lens kits, respectively, were assembled strictly according to specifications with silicon rubber RTV5 and polyurethane GF to validate whether new adhesive and process could eliminate high failure rate.

Validation test

Validation tests were proceeded according to production outline and military environment test standards as follows: (1) preleakage inspection, (2) high temperature test at 65°C for 48 h, (3) low temperature test at −55°C for 24 h, (4) thermal shock test from 60°C/2 h to −45°C/2 h in three rounds, (5) bumping test at six gravitational acceleration. (6) functional vibration and lasting vibration test monitored at every moment (7) hot and damp test at 30−60°C/(95 ± 5)% RH, 240 h; (8) salt fog test 35°C, 5% NaCl fog for 240 h; (9) postleakage inspection not only with 0.7 atm but also with 1.0, 1.5, and 2.0 atm for 5 min. All optical kits had passed this inspection. No failure was found. It proved that improving measures were effective and their quality with the new adhesive was high and reliable.

CONCLUSIONS

From the aforementioned tests, the following conclusions can be drawn:

1. The combined stress effect arose from unsufficient released crosslink stress for high strength and rigid epoxy adhesive and from thermal stress because of different expansion or shrink-

age of optical parts, while temperature changed drastically in environment condition tests. Optical lens with poor shear and tensile strength could not bear the combined stress resulting in failures. This was the major cause that resulted in high failure rate.

2. Failure rate at D production is obviously higher than do previous stages, because the deposited time for this stage is too short to release enough stress.
3. Assemblies that passed military inspection should be reliable and must meet the requirements of the coming trial test.
4. PU-2 polyurethane adhesive and RTV560 silicon rubber adhesive are suitable for bond optical kits working at aviation conditions, with sharp temperature changes and rigorous vibration. They can meet the requirements of some weapon system reliably.
5. Failures can be greatly eliminated by bonding the joints with high flexibility, low solidification stress, and good strength structure adhesive, such as PU-2 and RTV560, and controlling the minimal match clearance.
6. Area cleaned with solvent should dry adequately. Preparing, degassing, and employing operation of polyurethane adhesive should strictly follow specifications, cured at 70°C for 1–2 h, to guarantee the gum layer to attain the best function.

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