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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 074-0188

1. AGENCY USE ONLY (Leave blank)

2. REPORT DATE
April 1994

3. REPORT TYPE AND DATES COVERED
Stage 4 report, 1994

4. TITLE AND SUBTITLE
Laser Induced Reaction for Prebond Surface Preparation of Aluminium Alloys

5. FUNDING NUMBERS
Contract number F61708-93-C005

6. AUTHOR(S)
M. Rotel, J. Zahavi, A. Buchman and H. Dodiuk

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Israel Institute of Metals, Technion,
Haifa, Israel

Materials and Processes Department,
RAFAEL, Haifa, Israel

8. PERFORMING ORGANIZATION REPORT NUMBER
N/A

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

SERDP
901 North Stuart St. Suite 303
Arlington, VA 22203

10. SPONSORING / MONITORING AGENCY REPORT NUMBER
N/A

11. SUPPLEMENTARY NOTES
Prepared by Israel Institute of Metals, Contract number F61708-93-C005. This work was supported in part by Contract number F61708-93-C005.

12a. DISTRIBUTION / AVAILABILITY STATEMENT
Approved for public release: distribution is unlimited

12b. DISTRIBUTION CODE
A

13. ABSTRACT ***(Maximum 200 Words)***

The objective of this research is to establish the effect of excimer ArF UV laser on the Al alloy surface microstructure and activity and to find its correlation with the macro behavior of shear strength and failure locus. The system treated was adhesively bonded Al joints with structural adhesives. These adhesives are normally used in bonding and repairing processes for aerospace application. Surface treatment for bonding Al adherends with structural adhesives involve the sue of harsh chemicals such as acids, bases, and organic solvents. Laser surface irradiation can therefore be used as an alternative, ecologically favorable treatment. In order to achieve high adhesive strength optimal laser parameters for the treatment should be chosen (repetition rate, energy and irradiation time).

14. SUBJECT TERMS
excimer ArF UV laser, Al alloy, SERDP

15. NUMBER OF PAGES
30

16. PRICE CODE
N/A

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
unclass	unclass	unclass	UL

MINISTRY OF INDUSTRY & TRADE INDUSTRIAL R & ADMINISTRATION
משרד התעשיה
110 - 1994

ISRAEL INSTITUTE OF METALS · מכון המתכות הישראלי

TECHNION RESEARCH AND DEVELOPMENT FOUNDATION LTD.
מוסד הטכניון למחקר ופתוח בע"מ

LASER INDUCED REACTION FOR PREBOND SURFACE PREPARATION OF ALUMINIUM ALLOYS

Stage four report (1/94-4/94)
Contract No. F61708-93-C005

by

M. Rotel, J. Zahavi
Israel Institute of Metals, Technion, Haifa, Israel

A. Buchman, H. Dodiuk
Materials and Processes Department,
RAFAEL, Haifa, Israel

Haifa, April, 1994

ADDRESS: TECHNION CITY, HAIFA 32 000 / TEL. (04)294474 כתובת: קרית הטכניון, חיפה 32 000 / טלפון
FAX.: 972-4-235103 :.פקס TELEX: 46406 TECON IL :טלקס

CONTENT

1. INTRODUCTION

The potential of UV lasers irradiation as prebonding treatment of Al-2024 alloy was proved in a previous investigation(1) using a modified epoxy adhesive(2).
Surface treatment of Al by excimer laser resulted in homogenous oxidation, morphological changes and cleaning of the surface, promoting strong and durable adhesion. When applying optimal laser conditions (wavelength, energy, repetition rate and duration of irradiation) high adhesion shear strength was attained. This adhesion strength, achieved as a result of the laser treatment, is similar or higher than chemically treated Al (chromic unsealed anodization).

The objective of this research is to establish the effect of excimer ArF UV laser on the Al alloy surface microstructure and chemical activity and to find a correlation with the macro behavior reflected in shear strength and failure locus. The treated Al was adhesively bonded with structural adhesives.
The structural adhesives are normally used in bonding and repairing processes for aerospace application.Surface treatment for bonding Al adherends with structural adhesives usually involve the use of harsh chemicals such as acids, bases and organic solvents.Laser surface irradiation can, therefore be used as an alternative, ecologically favorable treatment. In order to achieve high adhesive strength optimal laser parameters for the treatment should be chosen (repetition rate,energy and irradiation time).

The fourth stage of this research (0004 of the contract) is summarized in the present report. This stage includes the results of tensile and peel tests of laser treated aluminum specimen and initial durability tests done by exposure of the specimen to heat/humidity and to extreme temperatures.

2. EXPERIMENTAL

2.1 Laser Treatment

The laser used during the course of this investigation is a UV excimer ArF (193 nm) laser EMG 201 MSC manufacture by "Lambda Physik", Germany. Beam cross section is 20mmx5mm with energy of 200mj/p*cm^2.Higher laser energies were achieved by reducing the laser beam area using focusing lens. Repetition rate was 30Hz and the number of pulses ranged between 1-5000. Specimen scanning is done by moving the specimen by means of a controlled x-y-z table. All experiments are conducted at ambient temperature and room environment. Fig 2.1 in the second stage report showed a schematic drawing and photo of the irradiation system.

2.2 Adherend and Adhesives

The adherend used throughout this work was an Al 2024-T3 alloy. The irradiated specimens were bonded by three different structural adhesives after primer application. Table 2.1 summarizes the data of the adhesives and the primers.

During this stage we received the primer BR154 which according to the data sheet is suitable for bonding with the adhesive FM350NA. Single Lap Shear (SLS) joints were primed and bonded with this combination.

FM350NA is a structural adhesive suitable for thermally high performance structures.

Table 2.1:The structural adhesives and primers

COMMERCIAL NAME (CYANAMID)	CURING CONDITIONS	APPLICATION FORM	SERVICE TEMPERATURE RANGE
FM73	1 Hr. 120 ^oC 40psi	FILM,0.38mm POLYESTER CARRIER	-55 ^oC to +120 ^oC
FM3002K	1.5Hr. 120 ^oC 40psi	FILM,0.3mm POLYESTER CARRIER	-55 ^oC to +175 ^oC
FM350NA	1Hr. 177 ^oC 30psi	FILM GLASS CARRIER	-65 ^oC to +177 ^oC
BR127 (chromate base)	1/2Hr. R.T 1/2Hr. 121 ^oC	MIXING,BRUSHING	-55 ^oC to +177 ^oC
A187 (silane base)	1/2Hr. R.T 1/2Hr.90 ^oC	BRUSHING 2cc A187 in 80cc ethanol and 20cc D.I. water	- NA -
BR154	1Hr. R.T 1Hr. 177 ^oC	BRUSHING	-55 ^0C to +177 ^0C

2.3 Testing

Adhesive joint properties were studied previously using SLS joints. In this stage the adhesive properties were studied using T peel joint according to ASTM D-3167 and FW tensile adherend joints according to ASTM C-297. Single Lap Shear joints (SLS) according to ASTM D-1002-72 were used to study the effect of extreme temperatures and heat/humidity (60^{0}c,95%RH) for 10 days on the laser treated bonded adherends as initial durability tests.

The mode of failure was determined to be either adhesive (locus of failure in the adhesive/substrate interface) or cohesive (locus of failure within the adhesive matrix), or mixed.

The surface of the irradiated area and the fracture surface morphology were studied by Scanning Electron Microscope (SEM) (Jeol model JMS 840, Japan) equipped with Energy Dispersive System (EDS, Link model 290).

2.4 Methodology

Two kinds of references are used in all the experiments: a non-treated Al 2024-T3 and an unsealed chromic acid anodized Al (according to MIL-A-8625C).The second reference is a conventional prebonding treatment for aluminum alloy.The strength of the reference joints were tested with the same adhesives and primers as the laser treated joints.

Primer application was carried out immediately after laser irradiation.

The adherends were kept in a desiccator between primer application and bonding.

3. **RESULTS AND DISCUSSION**

3.1 Tensile Tests

The FW joints were loaded in tensile mode at a rate of 1cm/min till failure. Tables 3.1 and 3.2 summarize the results of the tensile adhesive strength of the various structural adhesive joints. Figs. 3.1 and 3.2 show the failure surface of the joints with and without laser treatment.
The laser parameters for treating tensile joints were those chosen as optimal ones according to the SLS results (stage 2 report).SLS results for the combination of FM 350 NA adhesive and BR154 primer at various laser parameters is presented in chap. 3.4. The laser parameters for treating the tensile joint with BR154/FM350NA is the optimal one as presented in chap. 3.4 and varies from the parameters for FM73 and FM300-2K.

The results in table 3.1 show that laser treatment improves the tensile strengths in comparison to untreated specimen and attain values of 92% and 85% from the strength achieved with anodized specimens (for FM73 and FM3002K, correspondingly), For the adhesive FM350NA the tensile strength reached 89% of the strength of the anodized adherend.

Fig. 3.1 shows that the failure mode of the joints was totally cohesive for the adhesive FM73.For the adhesive FM3002K the laser treated and the anodized joints failed cohesively while the untreated primed joints failed adhesively (in the interface)(fig 3.1).The failure mode of the joints bonded with FM350NA and BR154 was cohesive for laser treated and anodized joints and mixed for untreated primed ones (fig.3.2).

Table 3.1: Tensile strength(Kg/cm^2) of non treated and laser treated FW joints. Laser energy $180mj/p*cm^2$, 2000pulses and primer A187.

Surface Treatment	Adhesive (FW)	
	FM73	FM-3002K
Without treatment	369±16(c)	113±12(A)
Anodized	430±8(c)	457±17(c)
Laser treated	395±18(c)	392±16(c)

Table 3.2: Tensile strength(Kg/cm^2) of non treated and laser treated FW joints. Laser energy $180mj/p*cm^2$, 600pulses and primer A187.

Surface Treatment	Adhesive (FW)
	FM350NA
Without treatment	158±31(m)
Anodized	289±21(c)
Laser treated	257±47(c)

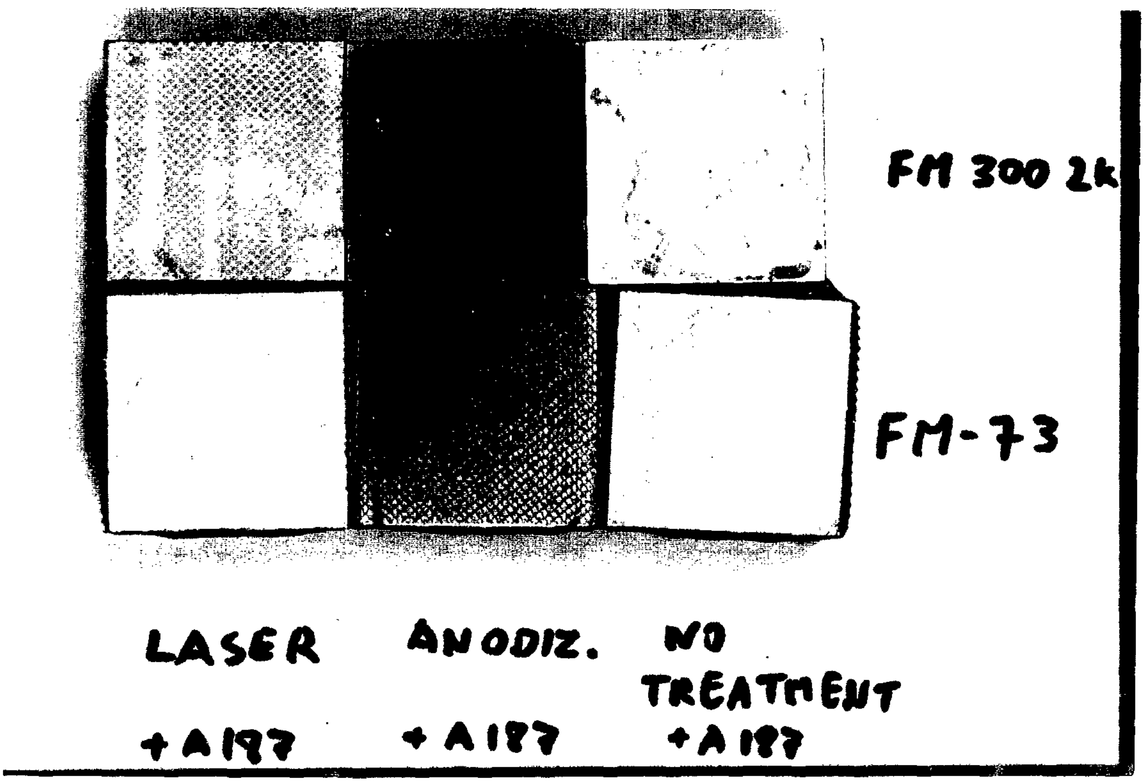
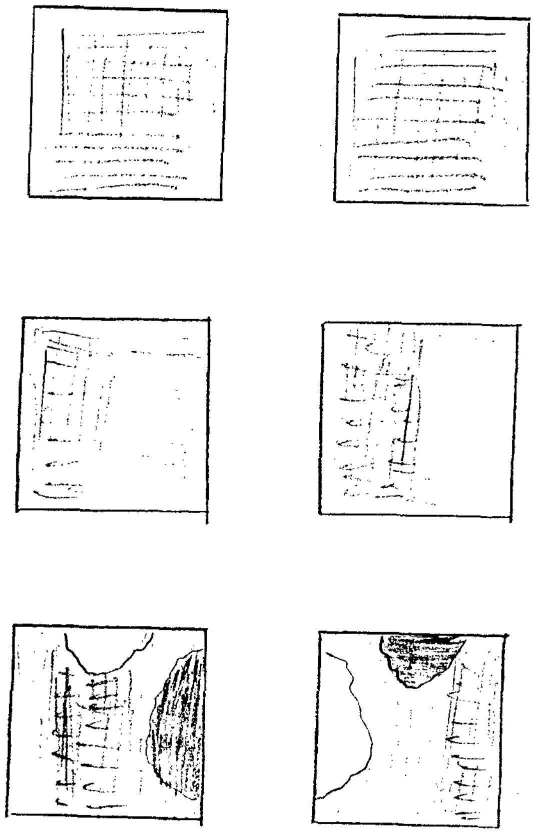


Fig. 3.1: Adherends after tensile tests.



Laser treated

Anodized

Without treatment

Fig. 3.2: Adherends after tensile tests.

3.2 T PEEL TESTS

Peel tests were conducted according to ASTM D-3167 at a rate of 200cm/min till failure. Tables 3.3,3.4 summarize the peel results of the various adhesives tests and fig. 3.3 show the adherends failure surface after the peel tests.
The specimens were irradiated at the optimal laser parameters defined from SLS tests. The irradiation of these specimens was done in a continuous scanning mode at a velocity corresponding to the step scanning used in stage 2.
The results in table 3.3 show that the resistance to peel of the laser treated joints was higher or the same as the anodized specimen for the adhesive FM73 and FM300 2K, correspondingly.
The resistance to peel of the laser treated joints was only 34% of the anodized specimen for the adhesive FM350NA and the primer BR154 (table 3.4) and twice that of the untreated primed joints. This results probably from the brittle behavior of this adhesive which shows low resistance to peel.

Fig.3.3 shows that the failure mode of the laser treated specimen was cohesive for FM73 and mixed for FM300 2K,similar to the failure mode of the anodized specimens.
The laser treated joints with FM350NA and BR154 failed adhesively while the anodized joints failed cohesively.

The highest resistance to peel was achieved for FM73 and its value was ten times that of FM350NA and FM300 2K,probably because the FM73 adhesive is more ductile.

Table 3.3: Resistance to peel (lib.inch) of non treated and laser treated joints (laser energy 180mj/p*cm^2 ,2000pulses,irradiation at continuous scanning at 2.7mm/min. ,30Hz) and primer A187.

Surface Treatment	Adhesive	
	FM73	FM-3002K
Without treatment	32.9±1.4 (97%c)	2.10±1.6 (100%a)
Anodized	31.8±3.3 (100%c)	4.56±1.0 (50%c)
Laser treated	37.2±1.7 (100%c)	4.42±0.2 (60-70%c)

Table 3.4: Resistance to peel (lib.inch) of non treated and laser treated joints (laser energy 180mj/p*cm^2 ,600pulses,irradiation at continues scanning at 8.9mm/min. ,30Hz.)

Surface Treatment	Adhesive FM350 NA primer BR154
Without treatment	0.63 (100%a)
Anodized	3.5±0.1 (100%c)
Laser treated	1.21±0.2 (100%a)

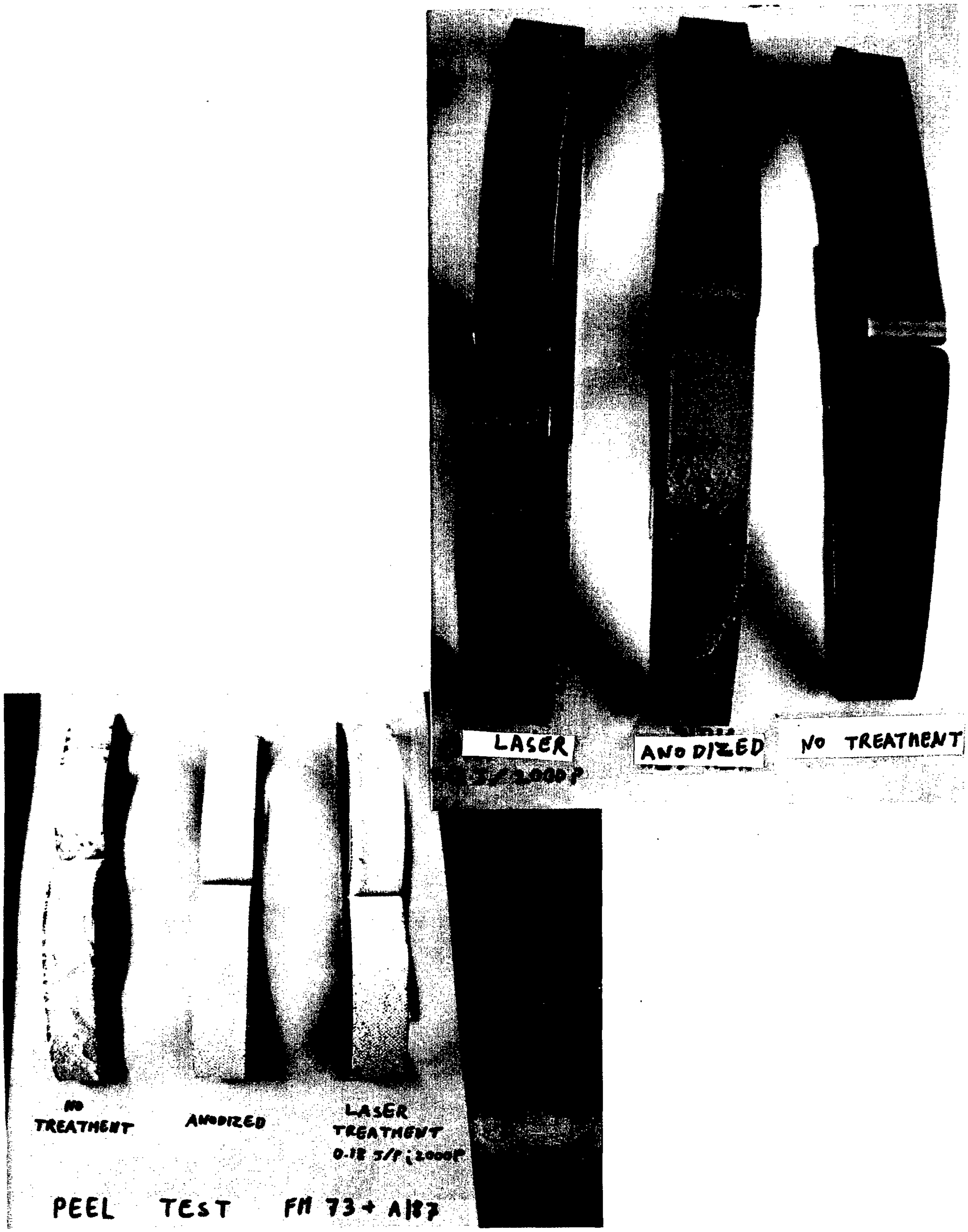


Fig. 3.3 Adherends after peel test.

3.3 Initial Durability Studies.

Initial durability studies of the resistance of laser treated bonded adherends in environmental conditions was done by measuring the shear strength after 10 days in humidity chamber (60^{0}c/95%RH) and by exposure to extreme temperature during SLS strength measuring.
Wedge tests will be conducted during the extension period of the project.

3.3.1: Joint Resistance to Humidity

The humidity resistance was tested on laser treated bonded adherends that were irradiated at optimal laser conditions, primed A187 and bonded with the adhesive FM73. These conditions were chosen because they revealed the highest shear strength, the highest resistance to peel and the highest tensile strength.
Table 3.5 summarizes the durability results, fig 3.4 shows the failure surface of the joints, and fig.3.5 shows the SEM observations of the morphology of the failure surface of SLS joints following exposure to hostile conditions.

The results show that the SLS strength of the laser treated adherends and of the anodized specimen did not change significantly after 10 days in humidity chamber in comparison to the untreated adherends joint which degraded by 27% in strength.
The mode of failure stayed cohesive after 10 days in humidity chamber for the laser treated adherends and the anodized adherends(fig.3.4). The untreated adhrends failed adhesively.
The failure surface morphology after humidity chamber (fig.3.5) did not present any changes in comparison to failure morphology before exposure (fig.3.12 stage 2 report).The failure was mostly cohesive revealing the net structure of the adhesive film carrier.

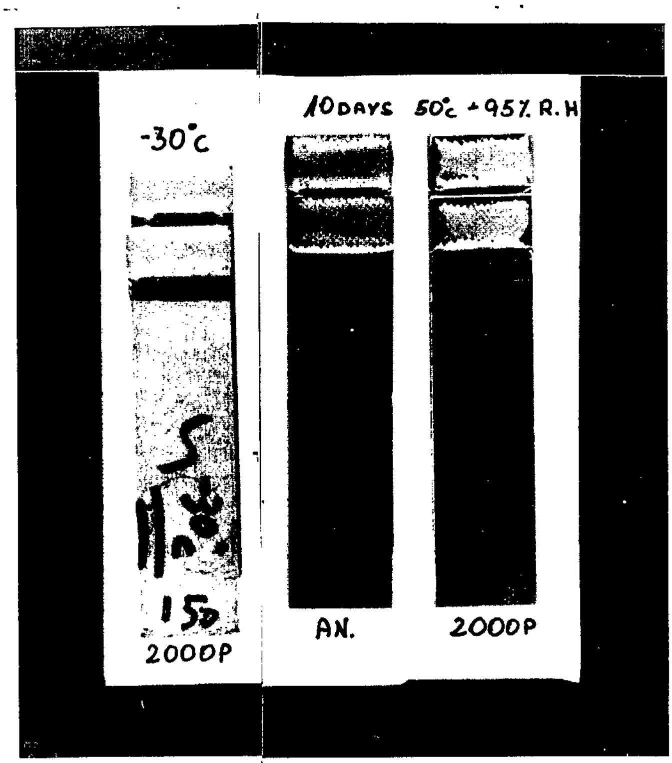
Table 3.5: Shear strength of laser treated adherends after 10 days in humidity chamber. Laser parameters: 180mj/p*cm^2, 2000pulses. Adhesive FM73, primer A187. Humidity chamber:10 days, 95%RH, 60^{0}C.

Surface Treatment	REFERENCE S.L.S [Kg/cm²]	AFTER HUMIDITY CHAMBER S.L.S [Kg/cm²]	CHANGE IN S.L.S %
UNTREATED (PRIMED)	285±35(M/A)	220±15(M/A)	-27
ANODIZED	394±18(C)	413±6(C)	+5
LASER TREATED	344±13(C)	320±36(C)	-6

C - cohesive failure

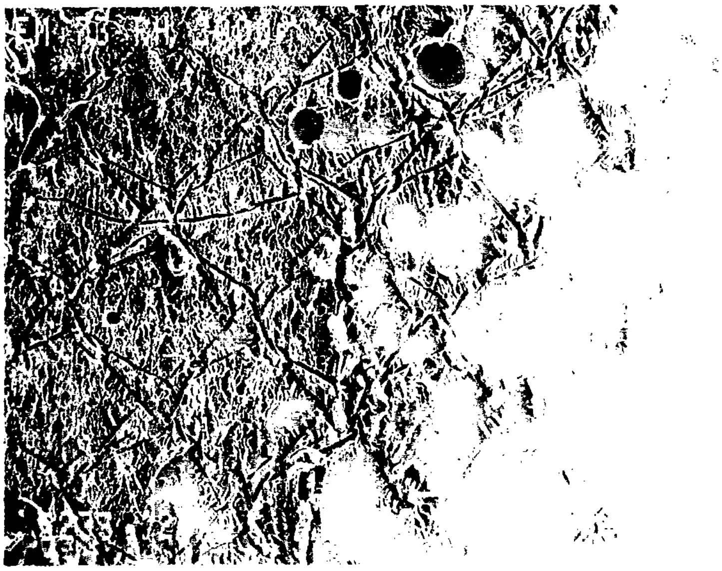
A - adhesive failure

M - mixed failure



B A

Fig. 3.4 SLS adherends: A) after 10 days in humidity chamber (95%RH,50^{0}C),and B) after testing at -30^{0}c. Adhesive FM73, primer A187.



General view

Adherence of the
adhesive to the substrate

Fig. 3.5: SEM photographs of the surface failure morphology of SLS joints after 10 days in humidity chamber. Adhesive FM73, primer A187. Laser energy 180mj/p*cm2,2000pulses).

3.3.2 : Shear Tests at Extreme Temperature.

The shear strengths at extreme temperature were tested on laser treated bonded adherends that were irradiated at optimal conditions primed with A187 and bonded with the adhesive FM73. These conditions were chosen because they revealed the highest shear strength, the highest resistance to peel and the highest tensile strength.
Table 3.6 summarizes the results of these tests,and fig 3.4 shows the failure surface of the joints.
The results show a significant improvement in shear strength at low temperatures (-30^{0}C) of the laser treated adherends. The shear strength increased by 40% compared to the shear strength at room temperature. In contrast, the shear strength of the anodized bonded adherends at -30^{0}C decreased by 16% in comparison to the shear strength at room temperature.
The failure mode at low temperatures was also cohesive(fig. 3.4). Due to the extreme high shear strength of the laser treated adherends at -30^{0}C (489Kg/cm^{2}) yielding of the adherends occurred (fig. 3.6).This phenomena was not observed for the anodized bonded adherends presenting lower shear strength (331Kg/cm^{2}) at -30^{0}C.
The shear strengths at $+90^{0}$C of the laser treated bonded adherends and the anodized bonded adherends decreased significantly compared to the shear strengths at room temperature. The locus of the failure changed from cohesive (at RT) to interfacial adhesive/substrate at high temperature. These results indicate the limits of performance of the adhesive FM73 with the primer A187 at this temperature ($+90^{0}$C). Although the values of the shear strengths reduced to 100Kg/cm^{2}, they still are adequate to the requirements for structural bonding.

Table 3.6: Shear strength at extreme temperature of laser treated bonded adherends. Laser parameters: 180mj/p*cm^2, 2000pulses. Adhesive FM73, primer A187.

Surface Treatment	R.T S.L.S Kg/cm²	-30°C S.L.S Kg/cm²	+90°C S.L.S Kg/cm²
ANODIZED	394±18(C)	331±40(C)	183±7(A)
LASER TREATED	344±13(C)	489±10(C) Al yielding	105±7(A)
UNTREATED	303±6(A)	---	72±14(A)

C- cohesive failure

A- adhesive failure

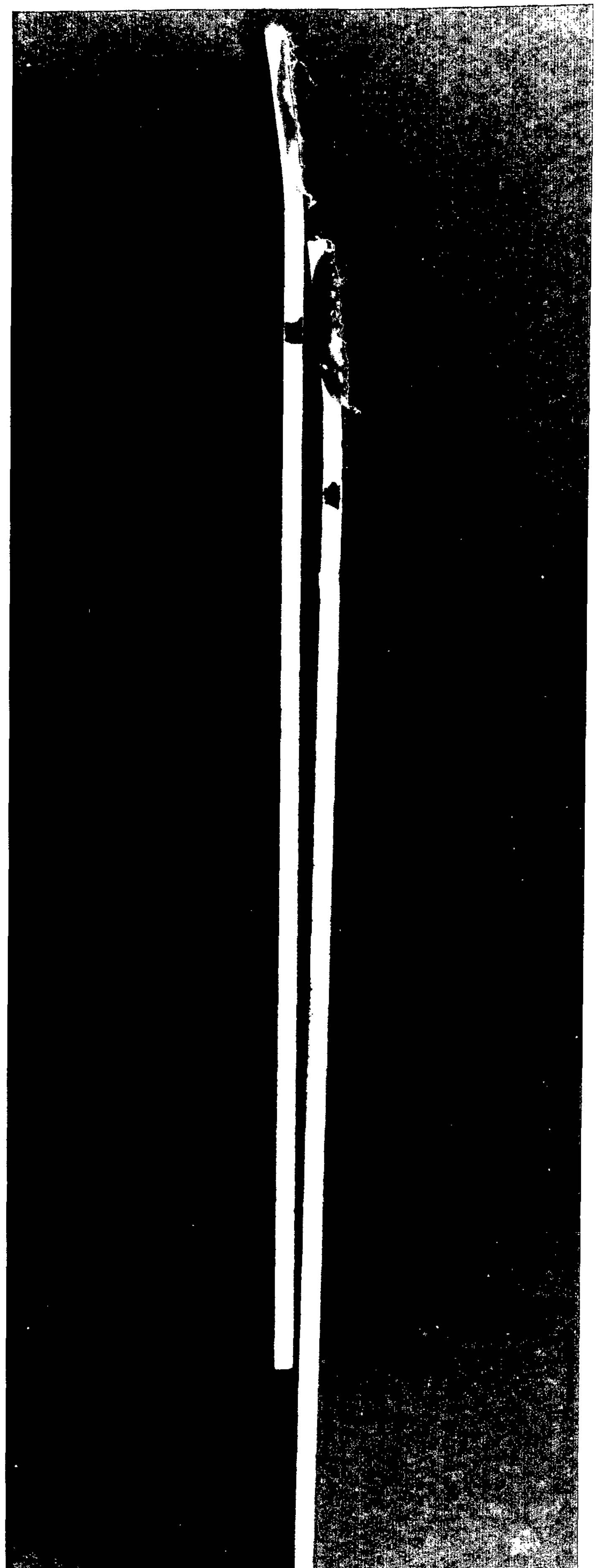


Fig.3.6: Visual observation of laser treated adherends after SLS test at low temperature.

3.4 SLS Tests with Primer BR154 and Adhesive FM350 NA .

SLS tests were performed in order to evaluate the effect of laser surface treatment on the adhesive bonding with the primer BR154 and the adhesive FM350NA. Table 3.7 summarizes the results of the SLS tests. The aluminum specimen were irradiated at various scanning velocity.

The highest shear strength obtained for the laser treated joints (table 3.7) was 66% of the shear strength of the anodized joints but better than the untreated joint. There values were lower than the values obtained for the other adhesives. Comparison with the results summarized at table 3.5 from the stage 2 report shows that using the primer A187 with the adhesive FM350NA resulted in higher shear strengths. The shear strength of laser treated joints was 217Kg/cm^2 with A187 and FM350NA and only 153Kg/cm^2 with BR154 and FM350NA. The anodized joints had shear strengths of 231Kg/cm^2 and 249Kg/cm^2 with FM350NA using the primers BR154 and A187 respectively.

The shear strengths of laser treated joints with A187 and FM73 or FM300 2K were also higher than that of FM350NA: 344Kg/cm^2 and 294Kg/cm^2 in comparison to 217Kg/cm^2, respectively.

The failure mode of the aluminum joints bonded with FM350NA was adhesive at the primer. This was also observed for the anodized adherends. These results indicate that the primer BR154 is unsuitable for the adhesive FM350NA as the failure occurs at the interface of the primer.

Table 3.9: Shear strengths of Al joints (adhesive FM350NA, primer BR154).

Surface Treatment	Laser energy at 30Hz mj/p*cm^2	Scann. velo-city mm/min	No. of Pulses	Shear Strength kg/cm^2	Failure Mode
Untreated				124+20	a(in primer)
anodized				231+37	a(in primer)
Laser treated	180	54	100	133±12	"
	"	10.8	500	149 ±8	"
		8.9	600	153±4	"
	"	5.4	1000	141±9	"
	"	2.7	2000	126±24	"

3.5 SEM OBSERVATION

Aluminum samples irradiated at various laser conditions were examined by SEM in order to study the effect of laser energy and number of pulses on the surface morphology. Auger and FTIR analysis were also conducted to complete the information and gain better understanding. Figs. 3.8 - 3.12 present SEM observation of the aluminum specimen (their auger depth profiles were represented at stage 2 report in chap. 3.3).

Fig. 3.8 shows the SEM observation of the untreated and laser treated (at $0.57J/p*cm^2$) aluminum specimen surfaces. The major change due to laser treatment was surface smoothing after irradiation of 50 pulses, and formation of cracks and material removal after 1000pulses . Irradiation at $0.18J/p*cm^2$ did not produce any changes on the surface, although cleaning and oxides layer formation was observed, at this energy level by the auger spectroscopy (figs.3.18,3.19 stage 3 report).
The auger depth profiles of Al specimens treated at laser energy of $0.57J/p*cm^2$ with 10 and 1000 pulses showed formation of oxide layers, thicker (about 900 $\mathring{A}$) than those produced at laser energy of $0.18J/p*cm^2$ (figs 3.20 stage 3 report).
Fig. 3.9 shows SEM observations of Al specimen treated at laser energy of $1J/p*cm^2$ with 10 and 100 pulses. Irradiation caused surface smoothing,disappearing of the machining lines and evaporation of intermetalic particles leaving small holes at the surface. Irradiation of 100 pulses resulted in formation of fine ripples on the surface(fig. 3.9b).
Auger profiles (fig.3.20 stage 3 report) indicated that irradiation with 10 pulses at $1J/p*cm^2$ resulted in formation of oxide layers of Al and Mg with thickness of about 700 $\mathring{A}$, thinner than the oxide layer produce at lower energy, probably due to ablation. Irradiation with 1000 pulses resulted in formation of aluminum oxide layer (without Mg). The relationship between Al and oxygen in this layer was similar to Al_2O_3 ,i.e. O: 60% and Al: 30%. The oxide layer thickness after 1000 pulses was about 600 $\mathring{A}$.

Fig.3.10 presents SEM observations of the surface morphology of specimen treated with laser energy of $2.7J/p*cm^2$ at 10, 50 and 100 pulses.

Irradiation at this energy level resulted in the disappearing of the machining line and creation of a rougher surface than that obtained at lower energy. Irradiation with 10 and 50 pulses resulted in a wavy morphology with embedded particles. Increasing the number of pulses to 100 resulted in smoother morphology with protruded ripples and holes originated from particle evaporation.

Auger depth profiles indicated that the combined reaction of ablation and melting resulting in introduction of nitrogen into the upper surface layer(Al nitration) in a very thin oxide -nitrogen aluminum layers (fig. 3.22 stage 3 report). The nitrogen content increased to 25-30% at depth of 60 $\overset{0}{A}$ and 20 $\overset{0}{A}$ (for 10 pulses irradiation and 50 or 100 pulses irradiation,respectively) and than decrease to 10% and less at a depth of 150 $\overset{0}{A}$. The oxygen content decreased from 30% at the surface to less than 10% at the depth of 150 $\overset{0}{A}$.

Figs 3.11,3.12 show the macro effects of laser energy on Al irradiation. At tower energy the surface topography did not change significantly (fig.3.11) while at $2.7J/p*cm^2$ the surface became rougher with deep laser indentations (fig.3.12).

The above results showed that different processes occur at various laser energies and time of irradiation. At low laser energy $0.18J/p*cm^2$ ablation of organic contamination and oxidation of Al and Mg occurred without morphological changes due to mostly photochemial ablation and photo-oxidation reactions.

A further increase in energy density caused a modification of the surface morphology. The threshold energy density for the first detectable modification correlates mainly with the thermal conductivity of the irradiated adherend. A low thermal conductivity causes earlier melting and smoothing of the surface due to the molten liquid.At high thermal conductivity (Al) a roughening was observed at energy density below $1J/p*cm^2$, and an oxide layer was formed on the surface.

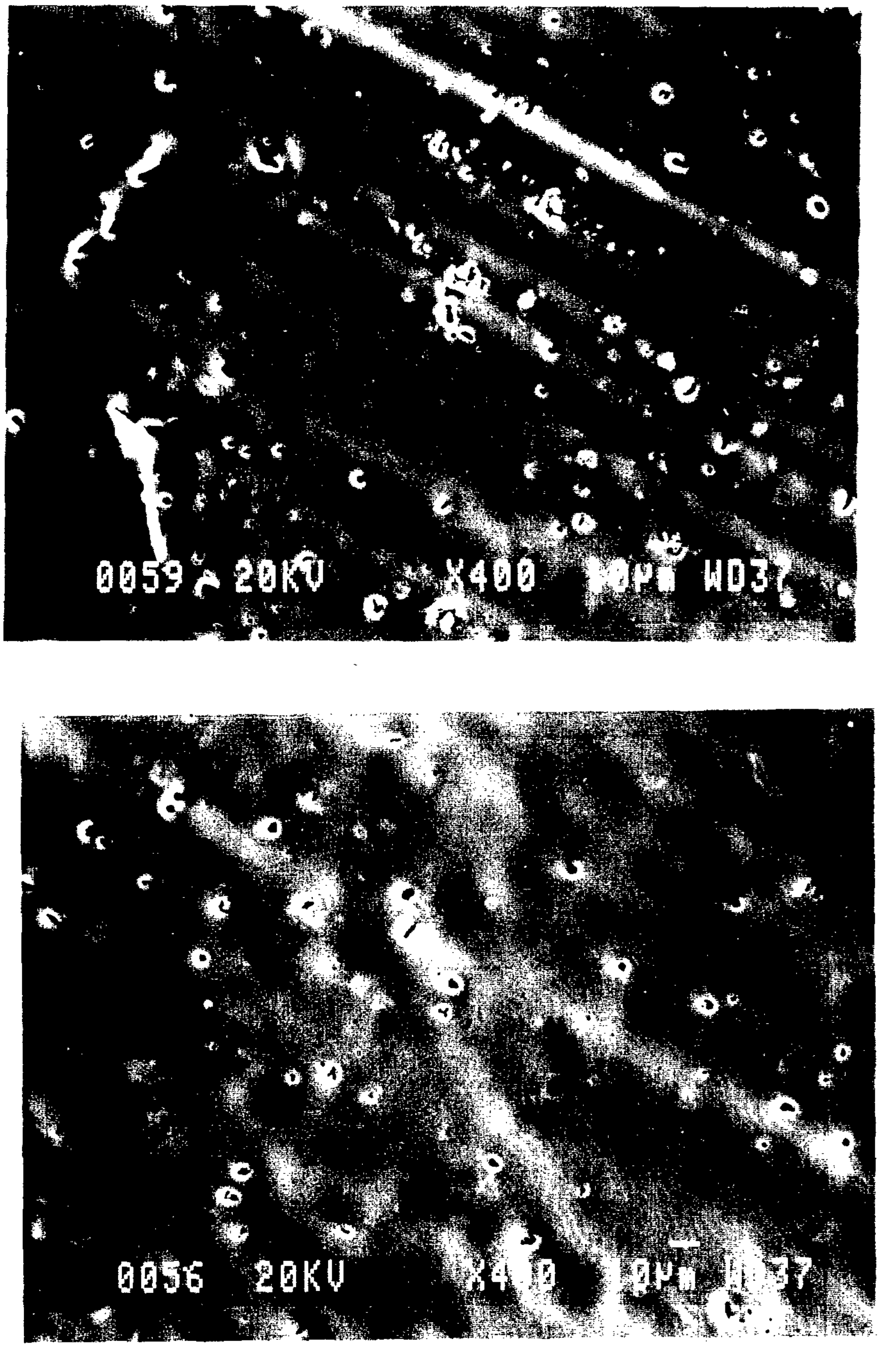
At $1J/p*cm^2$ the laser energy density was high enough to produce surface smoothing through thermal ablation. Higher laser energy ($2.7J/p*cm^2$) resulted in massive plasma formation and an increased surface roughness. This roughness resulted from an explosively spreaded plasma cloud that freezes on the surface in rapid solidification. The plasma wave moves from the middle to the rims as can be seen in fig.3.12. Because of the plasma formation additional reactions participate such as nitridation.

a

b

c

Fig. 3.8 : SEM photograph of Al surface: a) untreated, b)after irradiation with 0.57J/p*cm^2, 50 pulses, c)after after irradiation with 0.57J/p*cm^2,1000 pulses.



a

b

Fig. 3.9 : SEM photograph of Al surface: a)after irradiation with $1J/p*cm^2$, 10 pulses, b)after after irradiation with $1J/p*cm^2$,100 pulses.

a

b

c

Fig. 3.10 : SEM photograph of Al surface: a)after irradiation with 2.7J/p*cm^2, 10 pulses, b)after after irradiation with 2.7J/p*cm^2,50 pulses, c)after after irradiation with 2.7J/p*cm^2,100 pulses.

a

b

c

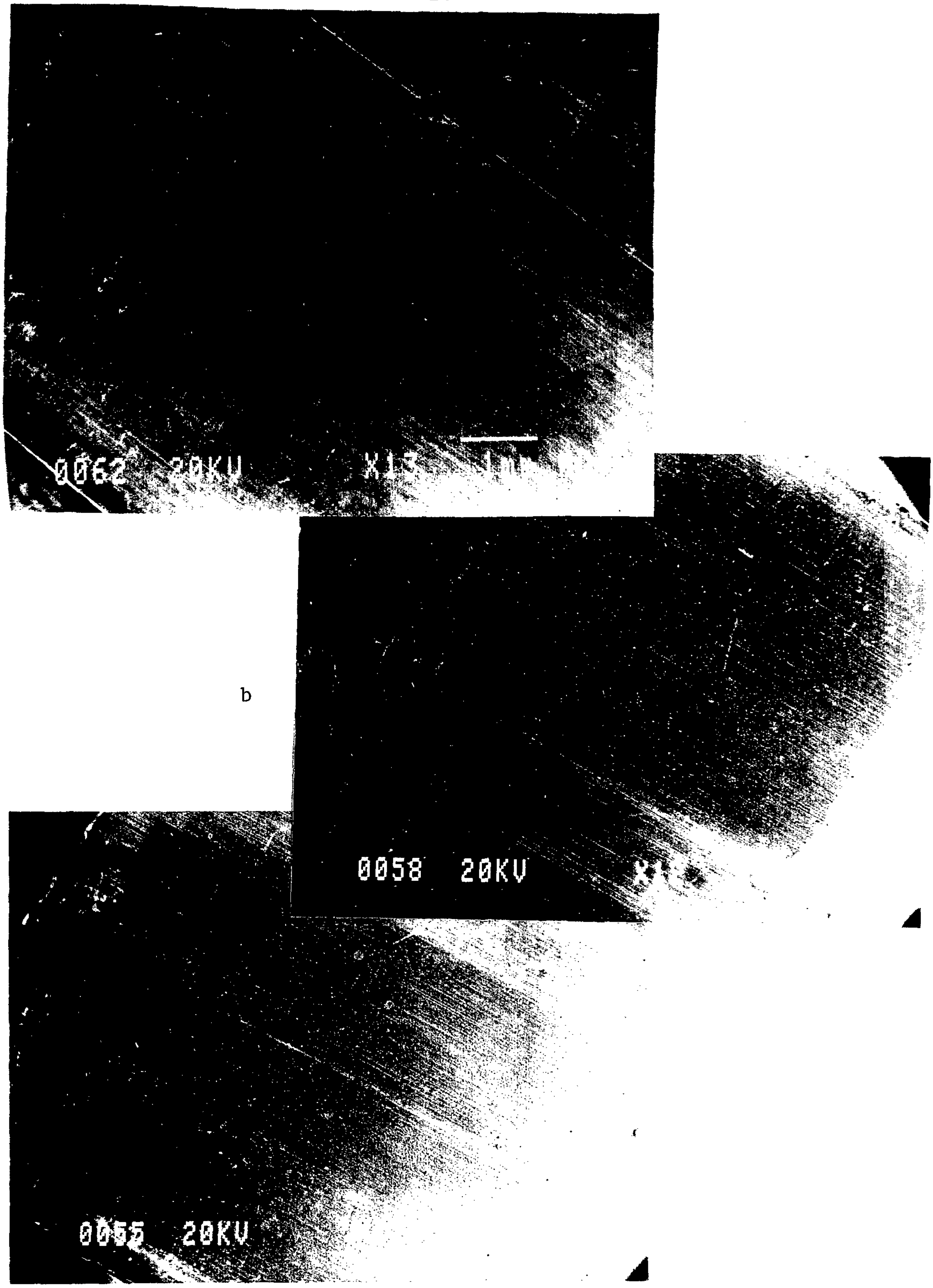


Fig. 3.11 : SEM photograph of Al surface: a)after irradiation with 0.57J/p*cm^2, 100 pulses, b)after after irradiation with 1J/p*cm^2,10 pulses. c)after after irradiation with 1J/p*cm^2, 100 pulses.

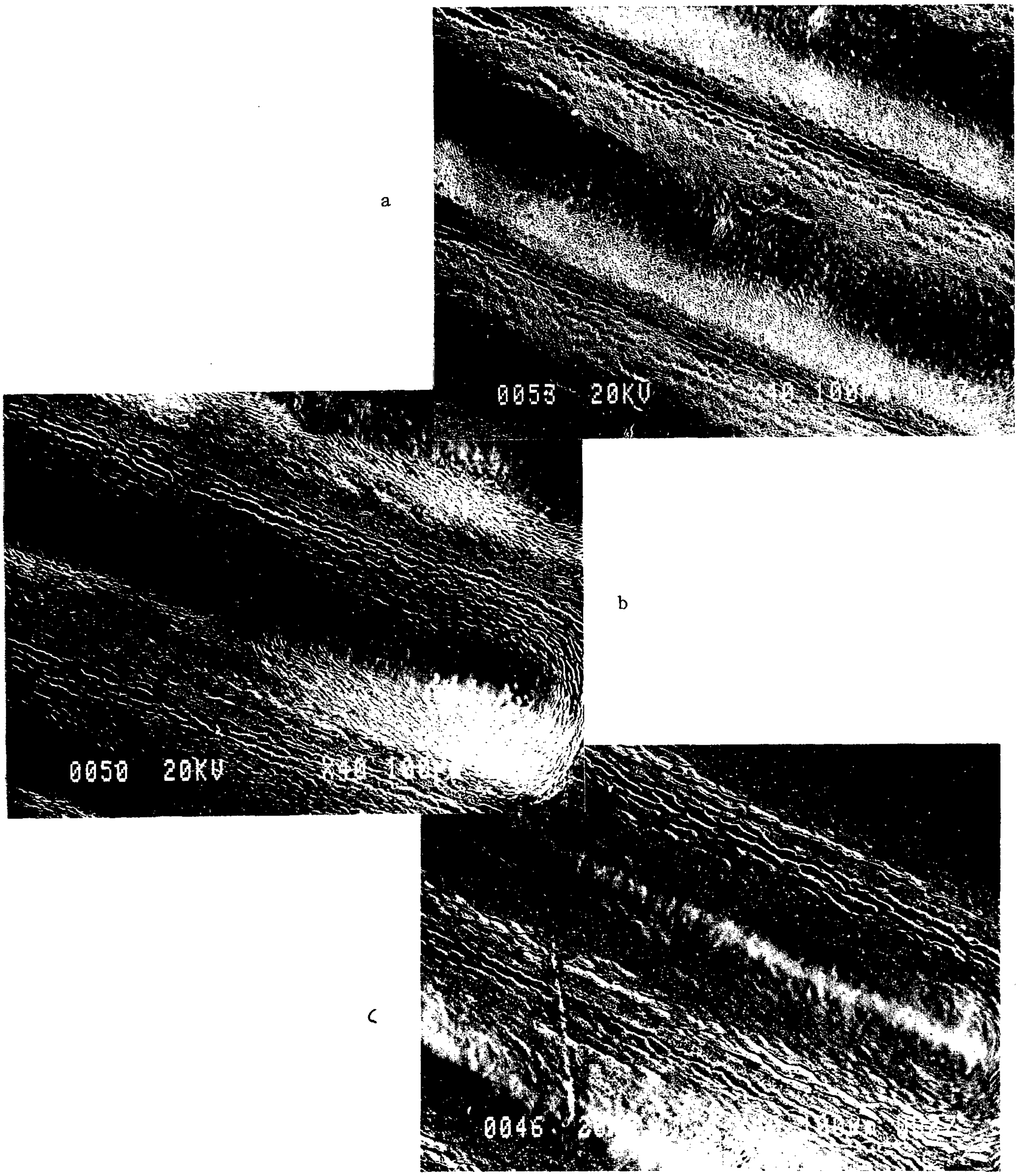


Fig. 3.12 : SEM photograph of Al surface: a)after irradiation with 2.7J/p*cm^2, 10 pulses, b)after after irradiation with 2.7J/p*cm^2,50 pulses, c) after after irradiation with 2.7J/p*cm^2,100 pulses.

4. SUMMARY

The forth stage of this research completed the mechanical tests for the three structural adhesives (FM73, FM350NA and FM300K) with Al 2024 adherends. T peel and tensile tests were produced.

Results showed that laser treatment of Al adherends with optimal laser parameters and priming with a silane primer A187 resulted in better adhesion strength than primed non treated joints. Adhesion strength was close to that obtained with anodization.

For all the adhesive tested, failure mode after laser treatment was cohesive or mixed which indicates the superior adhesion at the interface.

Durability tests showed that laser treated joints did not degrade in humidity chamber.

Testing at extreme temperature showed an advantage of the laser treated joints compared to non treated or anodized adherends.

SEM and Auger analysis indicated the various mechanisms involved in the laser treatment at different laser parameters (energy and time of irradiation).

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