EDGEWÖOD ARSENAL TECHNICAL REPORT

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VACUUM (OR FLUXLESS) BRAZING-GAS QUENCHING OF 6061 ALUMINUM ALLOY

by

M. M. Schwartz Advanced Manufacturing Technology Laboratory Martin Company Baltimore, Maryland 21203

Francis B. Gurtner and Paul K. Shutt, Jr. Technical Support Directorate

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Pre-Production Evaluation Division Technical Support Directorate EDGEWOOD ARSENAL EDGEWOOD ARSENAL, MARYLAND 21010

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FOREWORD

The investigation was started in January 1965; the initial phase has been completed, and advance work is progressing.

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Acknowledgments

The authors express their appreciation to Mr. R. O'Keefe and Mr. R. Donelson, Martin Company, and to Mr. G. Berg and Mr. F. Kirk, Jr., Technical Support Directorate.

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DIGEST

The purpose of this investigation was to provide a process or method for producing helium leak-tight containers under controlled processing and therefore with a high level of compatibility.

The concept evolved around combining vacuum brazing and gas quenching into one operation. The parts were quenched in the furnace after brazing or solution treating.

The advance into the field of vacuum brazing-gas quenching has provided a definite advance in the field of manufacturing for high or low volumes of items.

As a result of the investigation, the following conclusions were drawn:

1. Vacuum brazing-gas quenching is feasible for production quantities of complex and simple items.

2. The application of such a process is not limited to 6061 aluminum, but can be extended to ferrous and nonferrous alloys.

3. The future for a process such as vacuum brazing-gas quenching is unlimited at the present time.

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VACUUM (OR FLUXLESS) BRAZING-GAS QUENCHING OF 6061 ALUMINUM ALLOY

I. INTRODUCTION.

As more and more new metals are being developed, such as the superalloy and refractory-type metals, the potentials and capabilities of existing joining techniques are being challenged by present-day technology.

The culprit in most joining operations is contamination in one form or another. With the current interest in vacuum technology increasing, the authors decided to use vacuum brazing in an effort to control or eliminate the critical oxide contamination present with other joining techniques. Vacuum brazing eliminates the need for brazing flux, thereby lowering costs and excluding poor joint quality and corrosive damage, and eliminates the need for organic adhesives, which are often unsuitable at cryogenic or moderately high temperatures. There seems to be no reason why aluminum could not be vacuum-brazed and why the process could not be used (1) in brazing radiators and heat exchangers for aircraft and missiles, wave-guide fittings, and nuclear work; (2) in instances in which residual flux functionally poisons parts such as condensers and evaporators; and (3) in instances in which service requirements are too severe for organic adhesives or other joining materials and processes.

Gas quenching was tried because the authors wanted to maintain a high quality level in joining with respect to cleanliness, corrosion resistance, and compatibility. It was desired to eliminate water as a quenching medium and combine vacuum brazing and gas quenching into a single productive controlled function. Using this process, the postcleaning of assemblies is completely eliminated, and aluminum or steel tooling can be used instead of the more expensive stainless steel and nickel alloy tools.

II. EXPERIMENTATION.

A. <u>Alloy Selection</u>.

The aluminum alloys considered for vacuum brazing are those currently being brazed; namely, 2002, 3003, 6061, 2219, and 7005. The braze alloy was No. 718 (88% aluminum, 12% silicon), which is used in most brazing work today and usually in wire or foil form.

The initial tests were on lap joints and were conducted using nickel and silver plate for wetting. The results of these tests are described in table I. All failures were next to the lap joint. It was decided to examine the possibility of brazing without any coating.

Strip No.	Maximum load	Strip No.	Maximum load
	1b		1b
Ag-1	1,100	P-1	1,260
Ag-2	1,290	P-2	1,250
Ag-3	1,210	P-3	1,270
Ag-4	1,210	P-4	1,235*
Ag-5	1,140	P-5	1,255*
Ni-1	1.110	169-1	1,090
N1-2	1,230	169-2	1,265
N1-3	1,175	169-3	1,470**
Ni-4	1,105	169-4	1,185
·N1-5	1,050		

Table I.Results of Physical-Stress Tests on 0.125-in.6061 Lap-Joint-Brazed Strips

* Weld showed on Nos. P-4 and P-5; in all others, parent metal failed at edge f weld.

** Failed in grip at 1,365 lb; reran broken section; final break at 1,470 lb.

B. Cleaning and Initial Test Results.

The initial step in producing good brazed joints is to clean the aluminum surface. The cleanliness of the surface to be brazed is quite critical, and it was determined that brazing must take place within 12 hr after cleaning. The steps in cleaning are as follows:

- 1. Vapor degrease 5. Nitric-hydrofluoric acid dip
- 2. Alkaline clean 6. Water rinse
- 3. Water rinse
- 7. Oven dry at $230^{\circ}F$

4. Nitric acid dip

In addition, joint design, assembly tolerances, and general brazing practices that have been in use with either dip or furnace brazing, in which a flux was used to promote metal flow, were also adequate for vacuum brazing. The brazing time was 1 to 2 min, although allowance was made for the special heat-transfer problems inherent in a vacuum and for the capacity of the vacuum equipment to pump down to the proper limits. Brazing temperatures were 1,080° (minimum) to 1,100°F (maximum), with a ± 10 °F nominal tolerance range and a vacuum of 1 x 10⁻⁶ torr. Figure A-1* shows the interior of the vacuum furnace used. Initial tests were conducted on 0.040-in.-gage 6061 sheet material, which caused failures in the base metal; therefore, to achieve lap shear results, 0.125-in.-gage material was used. The test results are shown in table II. In addition, several dip-brazed and base-metal specimens were tested for comparison.

Type of braze	Dimensions	Area	Ultimate load	Strength stress
	in.	sq in.	15	psi
Vacuum Vacuum Dip Dip Dip Vacuum Vacuum	0.125 x 0.75 0.125 x 0.75 0.125 x 0.75 0.125 x 0.75 0.125 x 1.0 0.125 x 1.0 0.125 x 1.0 0.125 x 1.0	0.0937 0.0937 0.0937 0.125 0.125 0.125 0.125 0.125	880 930 955 1,030 1,218 1,147 1,260 1,155	9,400 9,800 10,200 11,000 9,750 9,180 10,080 9,230

Table	II.	Comparison	of Vacuum-	and Dip-Brazing	of
		0.125-in.	6061 Alumin	um Sheet	

C. Application.

With the encouraging results gained from test specimens, the process was applied to brazing actual hardware. Water was eliminated as a quenching medium because of its incompatibility with various reagents; an inert gas was substituted in order to achieve a T-4 condition in the base metal after brazing.

The first series of tests of time versus temperature was conducted using helium, argon, and liquid nitrogen. The test results showed helium to be the best. It was possible to lower the temperatures of the furnace and the part from the braze temperatures much more rapidly with helium than with any other gas. The dew point of helium and argon in all tests was -76° F minumum. All brazed and quenched bomblet canisters met the loak-rate criterion of 1 x 10⁻⁶ cc/sec for 15 sec (figure A-2).

The brazing clearance used was an 0.002-in.-interference fit. The maximum joint clearance was not determined.

* All figures, A-1 through A-23, are in the appendix.

The braze and heat-treat cycle consisted of:

1. Heat to 980°F and hold 30 min.

2. Raise temperature to 1,080°F and hold 30 sec.

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3. Introduce helium and cool.

III. DISCUSSION.

A. <u>Gas Quenching</u>.

The use of gas quenching for the manufacture of bomblet canisters is increasing because of the necessity to maintain a high quality level. The quality level is based upon three processing standards - cleanliness, corrosion resistance, and compatibility. When a bomblet is quenched by the common water method, it is impossible to remove completely the water and other compounds formed upon quenching. These residuals are the main cause of defectiveness in manufactured bomblet canisters.

The cleanliness level that is produced as a result of gas quenching does not, as of this writing, have a classification. Classifications for cleaning levels are going to be established in the near future. By cleanliness level, we are are implying that laboratory levels of cleanliness are being used under production conditions. Corrosion in this investigation refers to an acid buildup and subsequent[°] deterioration of the 6061-T6 aluminum alloy and a total loss of the part that has been under surveillance. Compatibility is the ability to store any compound inside the container under all types of conditions without any resultant reactions.

Figures A-3 and A-4 show two types of bomblet caristers that cannot be quenched by the water method. The maximum hole diameter for the passage of water is only 0.125 in.; it is in turn restricted by a deep well, which is only 0.060 in. from the 0.125-in. hole.

The gases first considered were helium, argon, and nitrogen, because of their relatively inert classification and lack of residual reaction with 6061 aluminum. Any oxidation of the radiant heating element would be kept to a minimum, as quenching would be accomplished in the furnace. The thermal conductivity of the three proposed gases is:

1. Helium - 3.32×10^{-4} Cal/sq cm/cm/sec/°C

2. Argon - 0.406 x 10^{-4} Cal/sq cm/cm/sec/°C

3. Nitrogen - 0.60 x 10^{-4} Cal/sq cm/cm/sec/°C

Of the three gases used for quenching from either the brazing temperature of 1,080° to 1,105°F or the solution-treating temperature of 980°F, helium has the best quench rate (figures A-5 to A-7). The curves illustrate the rates of quenching for both the furnace and the part. The most important curve is the one for the part. The first results from gas quenching were thought to be very good when the quenching rate was compared with the furnacetemperature charts, but the physical properties and hardness values depict a different story. The initial hardness values* for 6061 aluminum quenched by gases (utilizing 3/8-in. copper tubing to transfer the gas) are:

Argon	Helium

Ru	74	Ru	7	5,	.5	
- H	• •					

R_H 78.5 (15 days later) R_H 77 (15 days later)

The tensile strength was in the range of 23,000 to 24,000 psi. To facilitate better quenching, a 1-in. line was installed in place of the 3/8-in. line, and to obtain more data, thermocouples were attached to the parts. The change in the quenching medium and in the method of recording produced immediate results. The hardness values increased to a range of RH 79 to 80 for helium and R_H 78 to 79 for argon. According to The Metals Handbook, 8th edition, the hardness for 6061 in T-4 condition is $R_{\rm H}$ 80 to 102, with a minimum tensile value of 35,000 psi. The values given in table III show the reduced values. The designations refer to the following conditions:

- 1. MB1-1 furnace-cooled
- 2. MB2-1 helium-quenched
- 3. MB3-1 helium-quenched
- 4. MB4-1 - argon-quenched

0.5035

0.503

0.5035

0.503

0.504

0.5055

0.5025

5. EA - water-quenched (standard)

0.042

0.043

0.042

0.042

0.042

0.0425

0.042

of Brazed Strips as Received							
Designation	Width	Thickness	Area Load		Tensile strength	Elongation	
	in.	in.	sq in.	15	psi	%	

0.02115

0.02163

0.02115

0.02113

0.02117

0.02148

0.02111

435

550

545

490

795

795

790

20,565

25,425

25,770

23,190

37,555

37,010

37,425

21.5

21.0

22.0

20.0

22.0

22.0

24.0

Table III. Comparison of Tensile Strength

* Rockwell li.

MB1-1

MB2-1

MB3-1

MB4-1

EA-1

EA-2

EA-3

The hardness values (both $R_{\rm H}$ and R_{15T}) for samples MB1, MB2, MB3, MB4, and EA are given in table IV. The original goal of $R_{\rm H}$ 80 had been achieved (helium), even though the tensile strength did not reach 35,000 psi.

			H	ardnes	5				
Designation		Rockwell H					157		
		End 1			End 2				
MB1-1	68.5	69.5	69.5	70.0	71.0	70.0			
MB1-2	70.0	72.5	70.5						1
MB1-3	70.0	71.0	72.0				49.5	46.0	48.C
							ĺ		
MB2-1	81.5	81.0	82.5				1		
MB2-2	83.0	83.0	82.5				 		
MB2-3	81.0	80.5	81.0				58.0	57.5	58.0
							[
MB3-1	82.5	82.0	81.0						
MB 3-2	81.0	81.0	81.0						
MB3-3	80.0	80.5	81.0				61.0	57.0	57.0
							[
<u>УВ</u> 4-1	77.5	76.0	76.0						
MB4-2	77.0	78.0	78.0				l		
MB4-3	77.0	74.5	76.5				56.0	54.0	53.5
							1		
EA-1	94.0	94.0	95.0	96.0	97.0	95.5	1		
EA-2	95.5	96.5	96.0				Į		ĺ
EA-3	95.0	95.5	97.0						
EA-4	95.0	95.5	96.0				69.0	72.5	72.0
EA-5	96.5	96.5	96.5				73.0	73.5	74.0
		-					I		

Table IV. Hardness Values of Gas-Quenched Brazed Strips

B. Microstructures.

A survey of literature on the microstructure of aluminum alloys produced little information on isothermal curves and less on the T-6 condition, which is the final condition required for structural loading. With the considerable lack of information, two approaches were undertaken to gain more information: the development of isothermal curves and the determination of the metallographic structure of 6061 in the T-6 condition. The 6061 aluminum alloy, upon furnace cooling and gas quenching with argon and helium, was examined to provide an index for classifying the precipitate percentage. A percentage factor was related to each method of quenching so that an aging cycle could be developed proportional to the best conditions.

Operations to date have not permitted a thorough study of the structures and of their relationship to processing and heat-treating conditions. Previous conditioning of the material, which is described later, has a direct bearing on the final structure. In addition, 6061 aluminum alloy has a <u>memory</u> that cannot be removed through reheating.

The effects of air, argon, helium, and nitrogen quenching are illustrated by the microstructures in figures A-8 through A-13 (the aluminum was etched with sodium hydroxide).

Figure A-8 depicts the structure produced after the sample was brazed at 1,105°F and then cooled in the vacuum furnace. The same sample was used to determine and evaluate the effects of the gases and the heat-treating cycle upon the canisters.

Figures A-9 through A-13 show the structures produced by brazing, heat-treatment and quenching using varying methods.

Work is now underway for gas quenching bomblet canisters through several varying approaches. At present, retorts to contain the canisters and in which the gas-quenching operation can be applied are being investigated. Several interesting preliminary approaches are being tried: utilization of oxidizing gases and oxidizing and inert gases in combination; elimination of pressure gages; and altering the capacity and location of exhausts in order to attain a quench delay time of 2 min from a temperature of 980° to 0°F.

C. Solution Cycles.

To try for a greater hardness in the T-4 condition, other solutiontreating cycles were investigated, for instance, a double cycle.

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1. First Cycle

a. Step One: Anneal at 800°F for 2.5 hr.

b. Step Two: Increase temperature for solution-treating to 960° to 980° F and hold for 45 min.

c. Step Three: Increase furnace temperature to 1,080°F and hold for brazing.

d. Step Four: Gas-quench fr m brazing temperature.

2. <u>Second Cycle</u>

a. Step One: Increase furnace temperature to 960° to 980°F and hold for 45 min.

b. Step Two: Increase furnace temperature to 1,080°F and hold for brazing.

c. Step Three: Gas-quench from brazing temperature.

In the quenching operation for the first and second cycles, three large cylinlers were manifolded together and pressurized for immediate release into the furnace. The full volume from all three cylinders was expended on each furnace run.

Cycle No. 1 did not produce the hardnesses of R_H 85 to 30 that would have been anticipated by the additional time for annealing. The hardness value was lower using this cycle, and, therefore, the furnace runs were returned to the short cycle, No. 2.

To prove the feasibility of brazing and gas-quenching bomblet canisters under production conditions, several furnace runs were established per part volume. The volume of parts per run encompassed 25 to 200 units per furnace load without any relative loss in hardness or ultimate strength value. Figure A-14 shows the low-cost tooling for vacuum brazing-gas quenching 200 bomblet canisters at one time; a high or low volume was imposed for a continuous course of performances.

D. <u>Tolerances</u>.

In the day-to-day brazing controls, tolerances are expressed as loose, snug, or interferent, but rarely is a close control established for prepilot production. Several trials were made with fits between loose and snug (no interference) with good braze regults, but varying results as to meeting the helium leak criterion of 1×10^{-6} cc/sec for 15 sec. After several trials and a low yield, 80% per furnace run, corrections were made on one of the details that provided an interference tolerance of 0.002 to 0.004 in. between the mating parts that formed the lap joint of 1/8 in. The conformance to the tolerance of 0.002 to 0.004 in. produced a reliability of 97.6% from the brazing-quenching operation through a 100% 1 x 10⁻⁶ cc/sec helium leak test quality level.

E. Aging.

The standard aging cycle of 6 to 8 hr at 350° F was tried to determine the reaction of the bomblet canisters with a hardness of R_H 81. The results were poor, as almost no change took place.

At this point, several aging cycles were proposed, and four were tried for evaluation: 300° to 320° F for 67 hr; 300° to 320° F for 39 hr; $350^{\circ} \pm 10^{\circ}$ F for 8 hr; and $350^{\circ} \pm 10^{\circ}$ F for 6 hr, $190^{\circ} \pm 10^{\circ}$ F for 48 hr.

The results from three of the four aging cycles were encouraging for the use of gas-quenched units under actual test conditions. 1. Aging Cycle No. 1.

- a. Hardness as quenched in helium $R_{\rm H}$ 81 to 82
- b. 300° to 320° F for 67 hr R_H 88 to 96
- c. Ultimate tensile range 31,043 to 34,360 psi

d. Elongation, 13% to 13.5% (the low elongation corresponds to the low tensile value)

2. Aging Cycle No. 2.

a. Hardness as quenched in helium - R_H 81 to 82

- b. 300° to 320° F for 39 hr R_H 97 to 99
- c. Ultimate tensile range 34,000 to 35,000 psi
- 3. Aging Cycle No. 3.

No change.

4. Aging Cycle No. 4.

- a. Hardness as quenched in helium RH 81 to 82
- b. 350° ±10°F for 6 hr, 190° ±10°F for 48 hz R_P 93 to 98
- c. Ultimate tensile range, 33,333 to 35,000 psi

d. Elongation, 9.5% to 12.5% (the low elongation corresponds to the low tensile value)

Figure A-15 illustrates the class of microstructures that has been persistent throughout the investigation of gas quenching and aging. The microconstituents are distributed in a dispersion pattern and indicate a dispersion hardening effect upon the 6061 aluminum alloy (etched in sodium hydroxide). Further studies are underway to analyze and study the type of precipitate and its tentative mechanics of formation.

F. Quality Control.

The areas of investigation did not end with the development of an aging cycle, because the isothermal curve on the duplex aging cycle had been overlooked as to the effects. To initiate a proper duplex aging cycle, the uppermost temperature has to be lowered within 22 to 23 min and sloped to a temperature beyond the second cycle. Figure A-16 illustrates a typical duplex cycle: $350^{\circ} \pm 10^{\circ}$ F for 6 hr and $190^{\circ} \pm 10^{\circ}$ F for 48 hr. The cooling from 350° F

is accomplished by venting the furnace, resetting the temperature, and allowing a thorough circulation throughout the volume of material. Deviations from the temperature slope rates have resulted in material returning to a dead-soft condition.

The quality level of the gas is very critical, as a dew point of -85° F or bett r is a basic prerequisite. Beyond the dew-point stage, filtration equipment is now being installed and qualified for what compounds are being removed from standard grades of welding gas. Filtration has been successful in removing particles larger than 1μ (micron) by measuring the change in volume over a period of time. Qualitative and quantitative analyses have not yet been performed on the before and after results of filtration.

Measuring the quality level of the braze by nondestructive means has presently been reduced to using the helium leak test only. Figures A-17 to A-19 illustrate radiographs of vacuum brazed-gas quenched bomblet canisters. The radiographs are of two different densities. Figures A-18 and A-19 are of a similar density, and all but No. 5 passed the leak test.

Sectioning of those bomblet canisters that indicate filler metal not fully consumed showed that they still had a complete braze across the 1/8-in. joint.

G. Future Applications and Investigations.

and the second second

The future for the vacuum brazing-gas quenching process is very promising and will extend to items of varying sizes and complexity such as fuel tanks and rocket warheads, depicted in figures A-20 and A-21. In figures A-22 and A-23 are some other typical applications of vacuum aluminum brazing.

Some immediate advantages are:

1. Brazing flux is not used, and this provides a significant cost reduction compared to conventional practices.

2. No entrapped flux residues; thereby, poor joint quality and corrosive damage, which has often resulted in defective brazed aluminum assemblies, are eliminated.

3. Postcleaning of assemblies is completely eliminated.

4. Aluminum or steel tooling can be used instead of the more expensive stainless steel and nickel alloy tools.

In the commercial field, the next possible applications include radiators for automotive and air conditioning services in addition to various types of thermal conditioning sandwich panels and heat exchangers.

Further investigation should be conducted in the following areas:

1. Volume of filler metal versus joint tolerance and length.

2. Improve joint designs.

3. Evaluate other braze filler alloys.

4. Develop better braze alloys for corrosion resistance and leak-tight requirements.

5. Invesitgate gases as to their effects upon quenching rate, quench rate versus volume.

6. Evaluate nonbrazeable alloys (7075, 2014) and the joining of dissimilar combinations such as 6061 to 2219 and aluminum alloys to titanium, etc.

IV. CONCLUSIONS.

It is concluded that:

1. Vacuum brazing-gas quenching is quite feasible for production quantities of complex and simple items.

2. The application of such a process is not limited to 6061 aluminum alone, but can be extended to ferrous and nonferrous alloys.

3. The future for a process such as vacuum brazing-gas quenching is unlimited at the present time.







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Figure A-2. Vacuum-Brazed, Gas-Quenched Bomblet Canister That Met Helium Leak Criterion of 1 x 10⁻⁶ cc/sec for 15 sec Eikithteren dit armitet fettigt

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Figure A-4. Vacuum-Brazed, Gas-Quenched Bomblet Canister of Different Configuration



Figure A-5. Quenching Rates for Both Furnace and Part Using Helium Presolution (Solution-treated at 980°F for 45 min; brazed at 1,080° to 1,105°F)

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Figure A-6. Quenching Rates for Both Furnace and Part Using Argon Postsolution (Solution-treated at 980°F for 45 min; brazed at 1,080° to 1,105°F)

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Figure A-7. Quenching Rates for Both Furnace and Part Using Argon Presolution (Solution-treated at 980°F for 45 min; brazed at 1,080° to 1,105°F)

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Figure A-9. Microstructures of 6061 Aluminum Solution-Trested at 980°F for 30 min, Brazed at 1,080° to 1,105°F, and Quenched in Helium

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Microstructures of 6061 Aluminum Solution-Treated at 980°F for 30 min, Brazed at 1,080° to 1,105°F, and Quenched in Helium Figure A-11.

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Figure A-12. Microstructures of 6061 Aluminum Solution-Treated at 980°F for 30 min, Brazed at 1,080° to 1,105°F, and Quenched in Nitrogen





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Figure A-16. Typical Duplex Aging Cycle: 350° ±10°F for 6 hr and 190° ±10°F for 48 hr



Figure A-17. Radiograph of Vacuum Brazed-Gas Quenched Bomblet Canisters

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Figure A-18. Radiograph of Vacuum Brazed-Gas Quenched Bomblet Canisters

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Figure A-20. Fuel Tanks, Future Applications for Vacuum Brazing-Gas Quenching Process



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Figure A-21. Rocket Warheads, Future Applications for Vacuum Brazing-Gas Quenching Process



Figure A-22. Other Applications of Fluxless Brazing

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UNCLASSIFIED

Security Classification			
DGCUMENT	CONTROL DATA	A - R&D	
(Security classification of fills body of abstract and i	indexing annotation mus	ist he entered when the overall report is classified)	
CO. Edgewood Arsenal		INCLASSIFICATION	
ATTN: SMUEA-TSPP			
Edgewood Arsenal, Maryland 21010		N/A	
3 REPORT TITLE			
VACUUM (OR FLUXLESS) BRAZING-GAS Q	UENCHING OF 60	061 ALUMINUM ALLOY	
• DESCRIPTIVE NOTES (1, - of report and inclusive date The work was started in January 19	.) 65. and advanc	ce work is progressing.	
S AUTHOR(S) (Last name, lirst name, initial)			
Schwartz, M. M., Gurtner, Francis	B., Shutt, Pau	ul K., Jr.	
S. REPORT DATE	7. TOTAL NO	D. OF PAGES 75. NO OF REFS	
March 1967	49	θ ο	
SE CONTRACT OR GRANT NO.	9. ORIGINATO	OR'S REPORT NUMBER(S)	
N/A	PAGD 40	095	
5. PROJECT NO	EAIR 40	60	
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11 SUPPLEMENTARY NOTES	12 SPONSORIA	NG MILITARY ACTIVITY	
None	N/A		
The purpose of this investigation we helium leak-tight containers under high level of compatibility. The concept evolved around combining operation. The parts were quenched treating. The advance into the field of vacuus advance in the field of manufacturit As a result of the investigation, t	was to provide controlled pro- ng vacuum brazz i in the furna im brazing-gas ing for high on the following o	e a process or method for producin cocessing and therefore with a sing and gas quenching into on ace after brazing or solution. a quenching has provided a definit or low volumes of items. conclusions were drawn:	ng
 Vacuum brazing-gas quenchi complex and simple items. 2. The application of such a can be extended to ferrous and nonfi 3. The future for a process s at the present time. 	ing is feasible process is not ferrous alloys such as vacuum	te for production quantities of ot limited to 6061 aluminum, but a brazing-gas quenching is unlimit	ted
14. KEYWORDS		Fluxless brazing	
Vacuum brazing	Helium	Quenching	
Gas quenching	Alloys	Bomblet canisters	
Aluminum alloy	Argon	Containers	
6C61 Aluminum alloy	Brazing	Metals	
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DD .50RM. 1473	49	UNCI ASSIFIED	
		Security Classification	