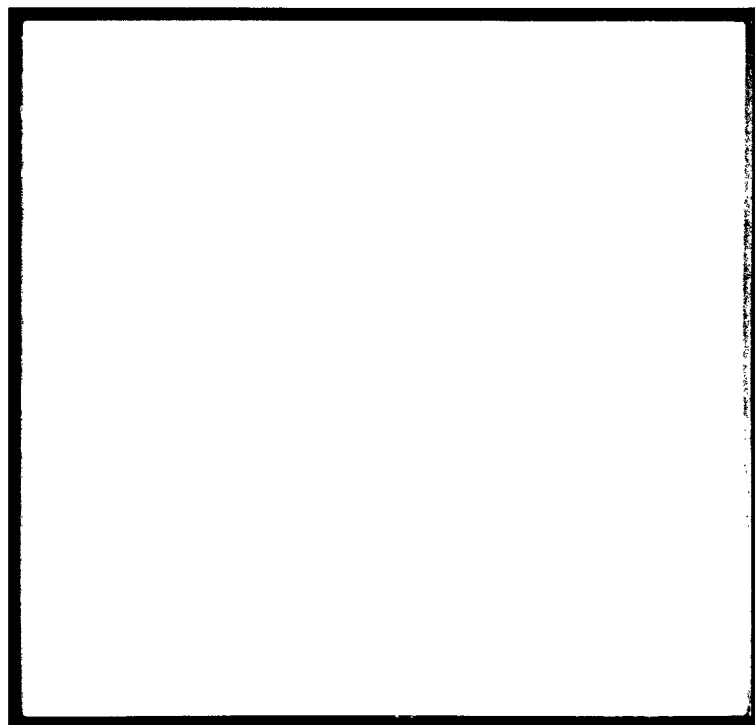


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INGERSOLL KALAMAZOO DIVISION
1810 North Pitcher Street, Kalamazoo, Michigan

ADHESIVE BONDING OF END CLOSURES
FOR HIGH STRENGTH AND RELIABILITY
IN ROCKET MOTOR CASES

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Prepared by:

D. P. Thomason
D. P. Thomason
Asst. Project Engineer

Approved:

L. E. Hershey
L. E. Hershey, Manager
Special Projects Department

INGERSOLL KALAMAZOO DIVISION
Borg-Warner Corporation
Kalamazoo, Michigan

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INTRODUCTION

Adhesive bonding of the forward and aft end closures to a tubular section offers several advantages over that of welded rocket motor case construction.

Uppermost among them are:

1. Increased reliability in high strength notch sensitive steels by eliminating welds and related contour and metal structure discontinuities.
2. Attachment of dissimilar metals without the normal problem of galvanic corrosion.
3. New fabrication possibilities such as combinations of steel cylindrical sections with heat resistant metallic or plastic aft closures and light weight metallic forward closures.
4. Better dimensional control of the parts during heat treatment due to elimination of locked up weld stresses and changes in section thickness.
5. Reduced cost by permitting each component to be finished prior to assembly, resulting in reduced size of heat treat and machining fixtures. Handling problems would be minimized and scrap would be reduced since it would be confined to the small relatively inexpensive components rather than the costly nearly completed assembly.

To realize these advantages of bonded case construction it would first be necessary to prove on a series of prototype units that adhesive bonded joints were physically capable of producing the high strengths required by this application. This, in essence, is the subject of this report.

Achievement of 3750 psi adhesive shear strength over each inch of a 4" long bonded joint for a total of 15,000 pounds per peripheral inch was the program goal. For most of today's missiles a bonded joint of this strength would exceed the strength of the case wall.

ABSTRACT

Seventeen sub-scale bonded joint cases of a 5" nominal diameter and 18" in length were fabricated and hydrotested. Fifteen of these units exceeded the target bonded joint strength of 3750 psi by an average of 40%. The information gained from the sub-scale cases was applied to the fabrication of eight prototype bonded end closure cases of 16" nominal diameter and 41" length. To assure failure of the bonded joint rather than the case wall, desirable for this program to permit direct examination of the bond line, the bonded joint length was decreased from the original 4" length used on the first five cases fabricated. Following the design change, prototype case number eight achieved the 3750 psi target joint strength.

Results of limited work with Super Tricent steel seamless and welded cases are also presented. This work was supplementary to that done with MBMC #1 welded cases in which controlled surface decarburization was used to reduce notch sensitivity.

SCOPE OF WORK

The scope of work authorized for this program consists of the design, fabrication and testing of high strength prototype cases having bonded rather than welded end closures. The objective is to determine the feasibility of this new fabrication technique. Prior to prototype case fabrication, experimental studies are to be conducted with bonded uniaxial specimens and bonded joint sub-scale cases.

In addition, limited work is to be done using MBMC #1 and Super Tricent steels for welded and seamless case fabrication. Controlled surface decarburization will be employed to reduce the notch sensitivity of these steels in accordance with laboratory specimen notch toughness data previously gathered on these materials. This work is in follow-up and conclusion to 1960 and 1961 decarburization studies.

MATERIAL

The prototype bonded end closure cases are to be fabricated from power spun Super Tricent steel forgings obtained from Taylor Forge and Pipe Works.

The sub-scale bonded joint test cases are to be made from cold drawn, aircraft quality, seamless AISI 4135 steel tubing.

Uniaxial specimens on hand from previous work are of both MBMC #1 and Super Tricent steel obtained by spinning forgings to the desired thickness, longitudinally parting and unrolling the tube and shearing out the specimens.

The high strength structural adhesive film which will be used to bond the uniaxial specimens, sub-scale test cases and prototype cases is FM-1000, 0.0045" thick, manufactured by Bloomingdale Rubber Company.

The welded and seamless cases will be fabricated from MBMC #1 and Super Tricent steels.

SUMMARY OF PROGRESS

I. Sub-Scale Bonded Joint Cases

Fabrication and testing of the sub-scale cases continued with test #6 and was concluded with test #17. The primary purpose of sub-scale case testing was to become familiar with procedures and problems of bonded joint case fabrication and to apply this knowledge to the fabrication of the larger prototype cases.

The sub-scale case testing program was divided into three parts which provided information pertaining to (1) scarf joint machining technique, (2) the effect of adhesive film thickness on joint strength, and (3) reproducibility of high strength results. A fourth important area of interest, bonding procedure, was not included as a primary subject of study in this phase of the project. This procedure, composed of joint cleaning technique, method of adhesive application, curing time and curing temperature was established prior to sub-scale case fabrication on the basis of previous experience of IKD research and development personnel. This basic bonding procedure, which proved satisfactory for the sub-scale cases, would be refined, if necessary, concurrent with and tailored for prototype case fabrication.

The design of the sub-scale cases is shown in Figure 1.

The Group I cases consisted of units 1 through 7, the first five of which have previously been reported. Sub-scale cases 6 and 7 were assembled, bonded and were hydrotested at the maximum pressure developed by the pump.

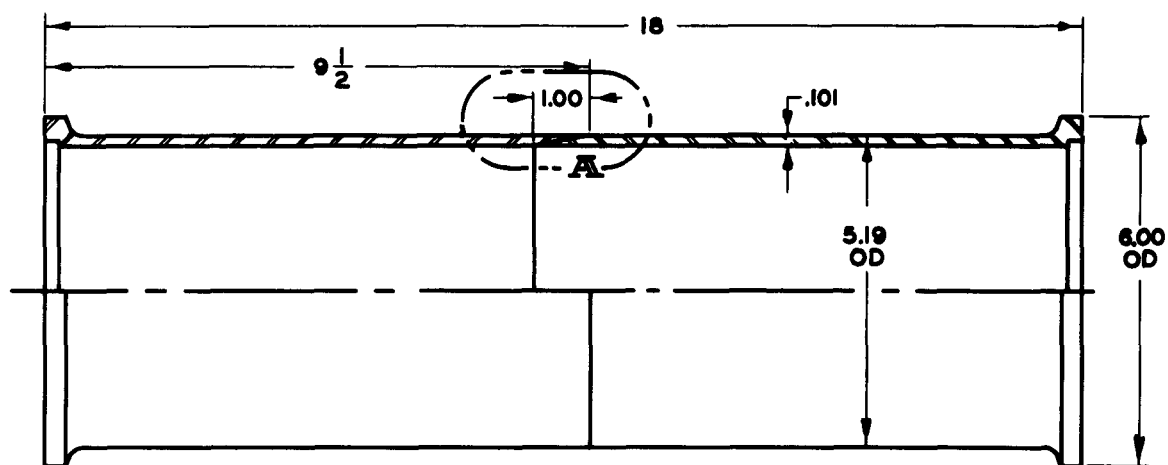
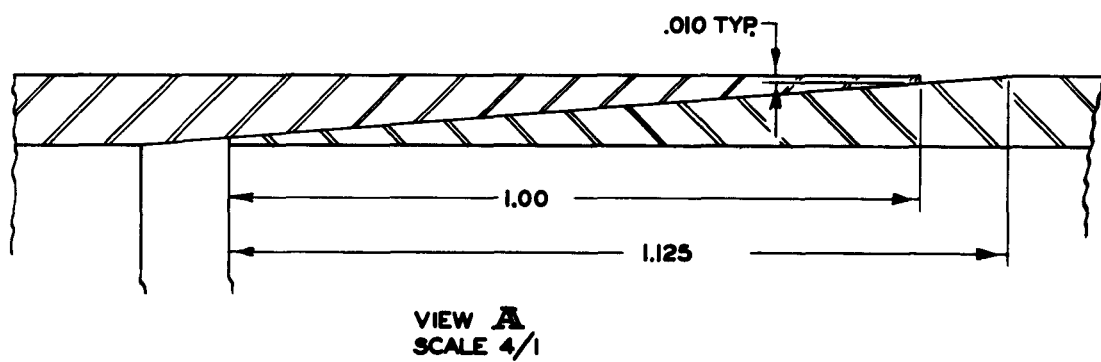


Figure 1. Sub-Scale Bonded Joint Test Case for Biaxial Loading



Pressure was held for three minutes at 4000 psig during each of two cycles, then was reduced and raised to 4800 and 4900 psig for cases #6 and #7 respectively and again held for three minutes. Neither case failed. Bonded joint strengths at the higher pressures were 6280 and 6300 psi. Inspection of each of the cases in this group during and after scarf joint machining and the results of the last two units particularly, had shown that the relatively simple rough machining and final grinding joint fabrication technique was satisfactory. Furthermore, it was evidenced that close joint tolerances with resulting good mechanical fits were reproducible using conventional machining equipment.

The number of layers of adhesive film used was the variable introduced in the fabrication of cases #8 through #12 representing Group II. Two layers of FM-1000 adhesive film (0.0045" thickness for each layer) were used throughout the Group I tests, with exception of case #1, therefore one layer was used on case #8 and three layers on case #9 and #10. To obtain a broader comparison between the effect of one and two layers of adhesive on bonded joint strength one and two layers of AF-15 adhesive film were used on case #11 and #12. Case #8, with one layer of FM-1000 adhesive, withstood hydrotest cycles of 4000, 4000 and 4800 psig, each pressure being held for three minutes. Bonded joint strength of this unit was in excess of 5990 psi. The strength of units #9 and #10, with three layers of FM-1000 adhesive, was 5430 and 3520 psi respectively. Some difficulty was experienced in maintaining bonding pressure on these two units during the cure cycle. As

the relatively heavy film glue line (0.0135" thickness) softened and excess adhesive squeezed from between the joints, the initial pre-set bonding pressure indirectly measured by longitudinal compressive force on the case, was gradually lost. It would have been possible to maintain this initial longitudinal force during the cure cycle by one of several means, however in so doing, more adhesive would be squeezed from the joint and the net result would have been the same as if one or two layers of film were used originally. Thus, it was found that there was no advantage to be gained by using more than two layers (0.009" thickness) of FM-1000 adhesive film in sub-scale case fabrication. The choice between the use of one or two layers of adhesive was still open, however. Using AF-15 adhesive film (0.003" thickness per layer), units #11 and #12 were bonded using one and two film thicknesses respectively. These units produced identical joint strength results during hydrotest, 4560 psi. This figure was quite near the ultimate strength of the adhesive. From the Group II tests it was determined that either one or two layers of FM-1000 adhesive would be suitable for use in prototype case fabrication.

The remaining tests, representing Group III, were conducted to establish reproducibility of high joint strength results. Five cases were machined, bonded and hydrotested. Due to temporary difficulty with the pressure system pump, maximum hydrotest pressure had to be limited to 4000 psig for these last cases. Four of the five cases did not fail during hydrotest. Case #14 which did fail after holding 4000 psig for one minute

was found to have traces of rust on the joint beneath the adhesive. This case was the only one of the 17 that had been assembled one day and cured the next. Evidently, rusting of the machined joint surfaces occurred prior to curing and resulted in unclean and correspondingly weakened joint of case #14. In spite of the moderately weakened joint of case #14 the joint strengths of the Group III cases were well over the target strength of 3750 psi and were consistent and reproducible. Table I shows the results of all seventeen sub-scale cases tested. Figure 2 is a photograph of a typical sub-scale case which has just been cured. Figure 3 is a photograph of case #7 which withstood a longitudinal force during hydrotest of 95,800 pounds with a bonded joint length of 0.96 inches.

II. Sixteen Inch Diameter Prototype Bonded Joint Cases

A. Case Design

The case design for the 16" nominal diameter bonded joint prototype cases is shown in Figure 4.

B. Case Fabrication Procedure

These cases were fabricated from hydrospun Super Tricent air melt steel forgings. Each spun case was slipped over a mechanical expanding holding and roundup fixture which was centered in a lathe and the end rings were cut from the tubular section. The lathe compound was set at 0.020" per inch taper and a 4" nominal length scarf joint was rough machined on the outside of the tube and the inside of the end rings. The rough machined case was then fixtured for heat treat and heat treated in an oxidizing

Table I. Sub-Scale Bonded Case Results

Case Number	Longitudinal End Force lbs.	Adhesive Film			Hydrotest Pressure psig	Joint Length Inches	Target Bond Strength psi	Actual Bond Strength psi	
		Type	Layers	Thickness					
1	300	FM-1000	1	.0045	2200	15/16	3750	2	2880
2	1700	FM-1000	2	.009	3000	15/16	3750		3920
3	5000	FM-1000	2	.009	4000 *	7/8	3750		5700+
4	5000	FM-1000	2	.009	4600 *	1.00	3750		5740+
5	5000	FM-1000	2	.009	3250	27/32	3750		4700
6	5000	FM-1000	2	.009	4800 O	61/64	3750		6280+
7	5000	FM-1000	2	.009	4900 O	31/32	3750		6300+
8	5000	FM-1000	1	.0045	4800 O	1.00	3750		5990+
9	5000	FM-1000	3	.0135	4000	59/64	3750		5430
10	5000	FM-1000	3	.0135	2600	59/64	3750		3520
11	5000	AF-15	1	.003	4000	1-3/16	3750		4560
12	5000	AF-15	2	.006	3200	7/8	3750		4560
13	5000	FM-1000	2	.009	4000 Δ	57/64	3750		5620+
14	5000	FM-1000	2	.009	4000	1-3/64	3750		4780
15	5000	FM-1000	2	.009	4000 Δ	1-3/64	3750		4780+
16	5000	FM-1000	2	.009	4000 Δ	15/16	3750		5330+
17	5000	FM-1000	2	.009	4000 Δ	1-1/32	3750		4840+

Notes: * Held for three minutes at pressure.

O Cycled at 4000 psig twice holding for one minute each time before raising to and holding at pressure shown.

Δ Held at pressure for one minute.

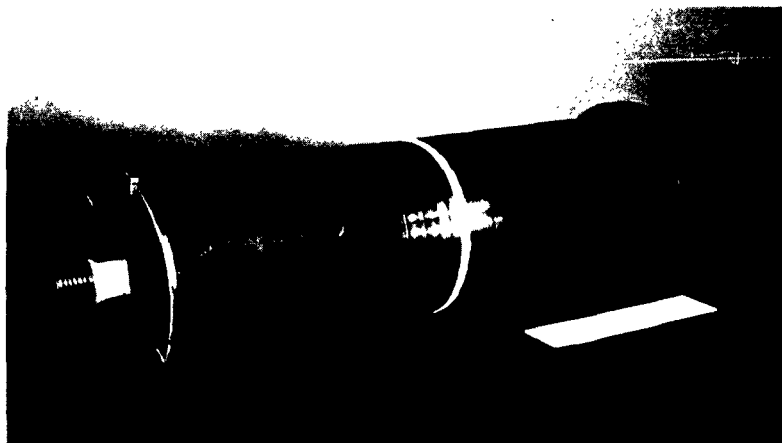


Figure 2. A Typical Sub-Scale Case
Which Has Just Been Cured

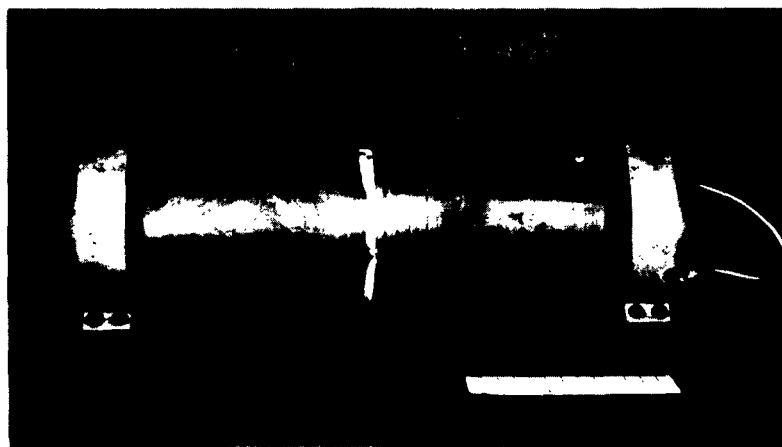


Figure 3. Sub-Scale Case #7. Hydrotest
cycled to 4000 psig, 4000 psig and 4900 psig
without failure. Bonded joint length was
.96" and withstood a longitudinal force of
95,800 pounds

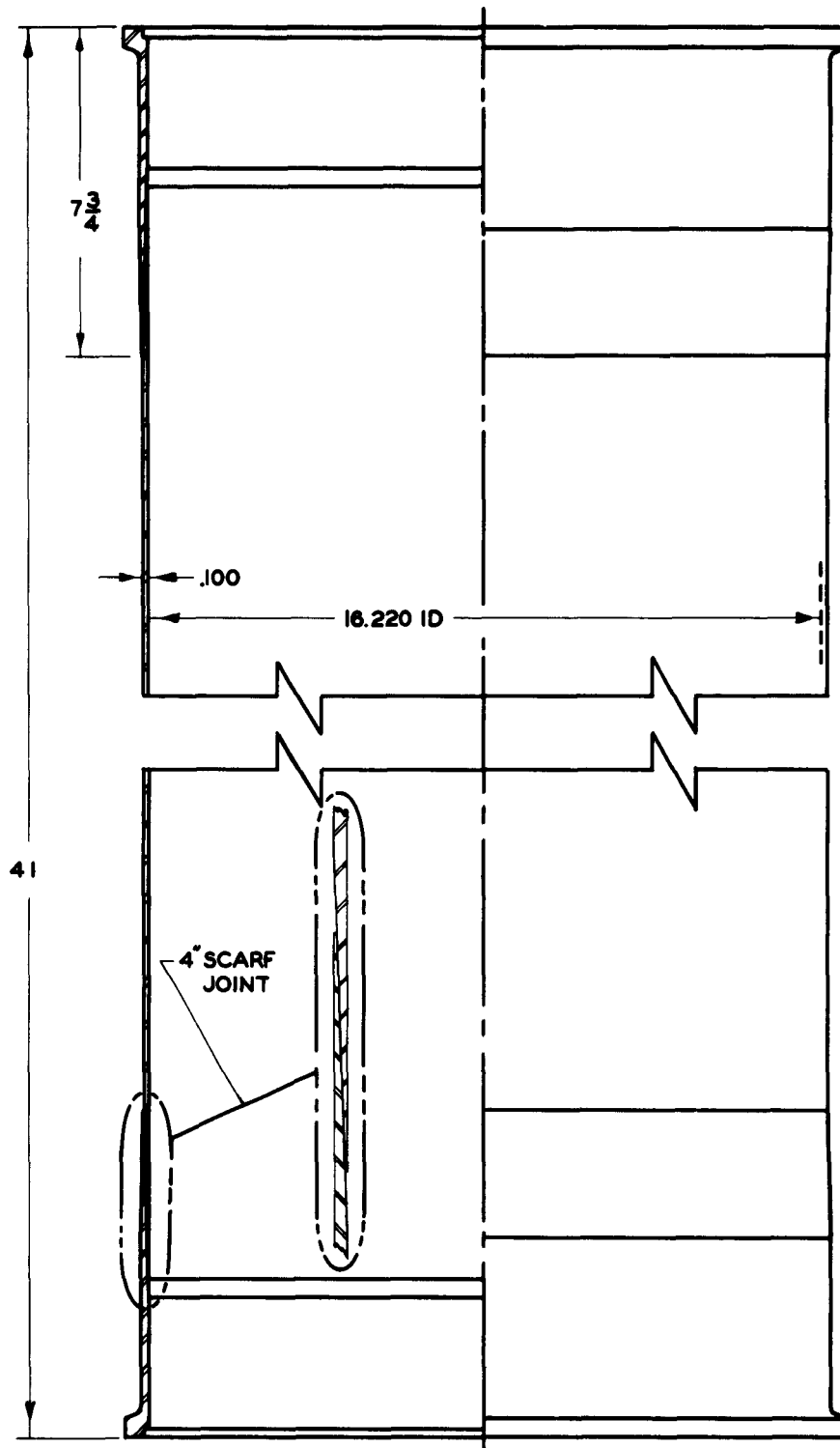


Figure 4. Basic Tube Design for Bonded End Ring Case

atmosphere to produce a given depth of surface decarburization and corresponding reduced notch sensitivity. The case was returned to the lathe and the joints were finish ground. Following grinding, the machined joints were thoroughly cleaned in preparation for adhesive bonding of the end rings to the tubular section. FM-1000 adhesive film was wrapped over the tapered tube ends and the end rings were reassembled to the tube and drawn firmly in place using a rod and end plate curing fixture and torque wrench. Finally, the assembled case was placed in the oven, thermocouples were attached and the adhesive cured. Figures 5 through 13 illustrate this fabrication procedure.

C. Discussion of Results

A total of eight cases were fabricated and hydrotested during this period. These eight cases comprised two groups of tests, the first five representing Group I and the latter three Group II. All variables including those pertaining to machining technique, assembly and curing were held constant within each group.

1. Group I Tests

As was done with the uniaxial specimens and sub-scale cases, this first group of tests was used to establish an average bonded joint strength from which improvements could be made as the program progressed. Secondly, it was intended that this first group serve to indicate any required changes or modification to be made in machining technique, case assembly procedure and/or tooling before proceeding to the Group II tests.

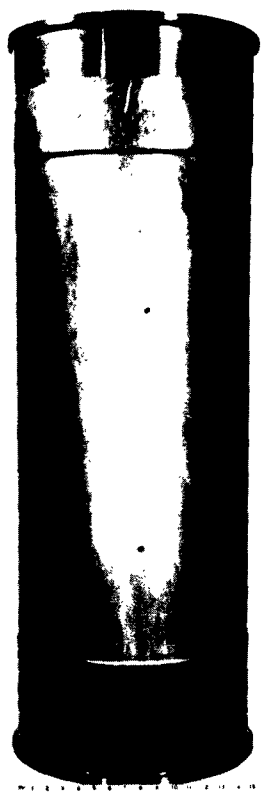


Figure 5. A 16" Diameter Power Spun Seamless Case from which the Prototype Bonded Joint Cases were Fabricated

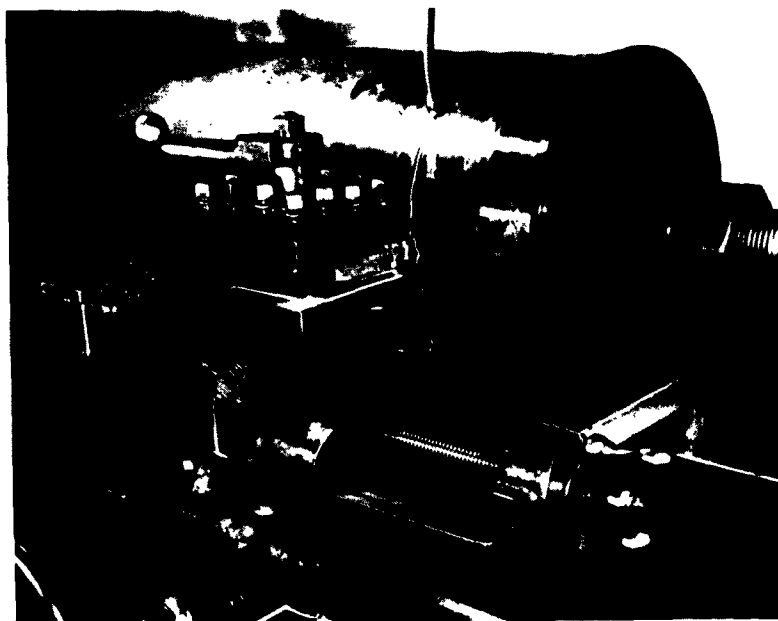


Figure 6. End Rings Being Parted from the Tubular Section of a Seamless Case

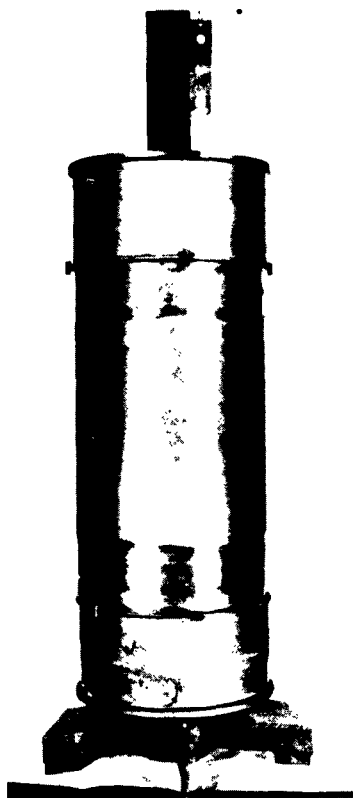


Figure 7. A Prototype Case Fixtured for Heat Treat Following Rough Machining of the 4" Scarf Joints

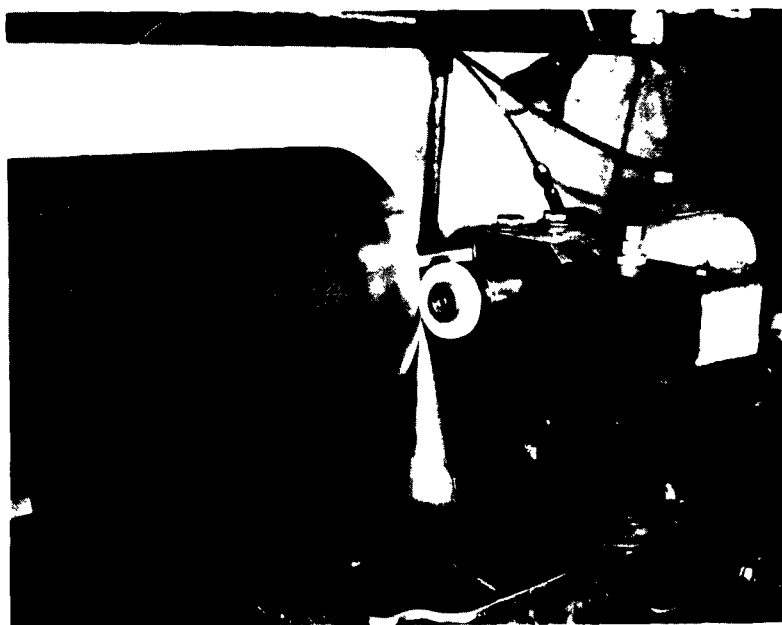


Figure 8. The Rough Machined Joints Being Ground After Heat Treat

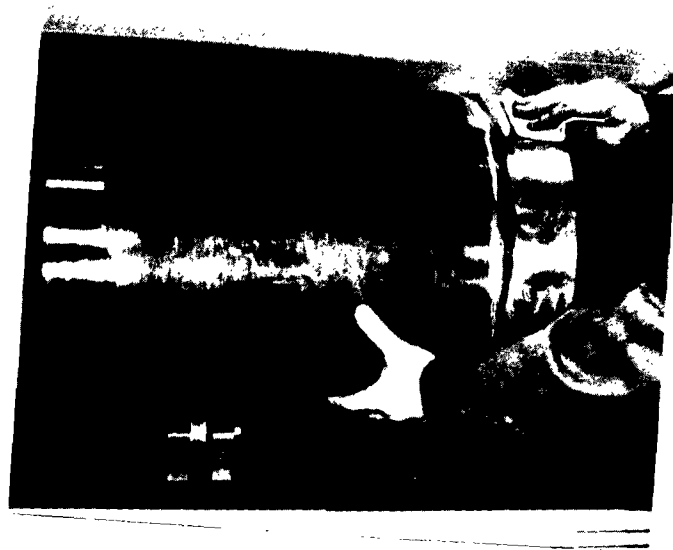


Figure 9. One Step in the Joint Cleaning Procedure



Figure 10. FM-1000 Adhesive Film Being Wrapped Over the Tapered Tube Ends



Figure 11. The End Rings Were Drawn onto the Tubular Section Using the Threaded Rod and End Plate Curing Fixture and Wrench.

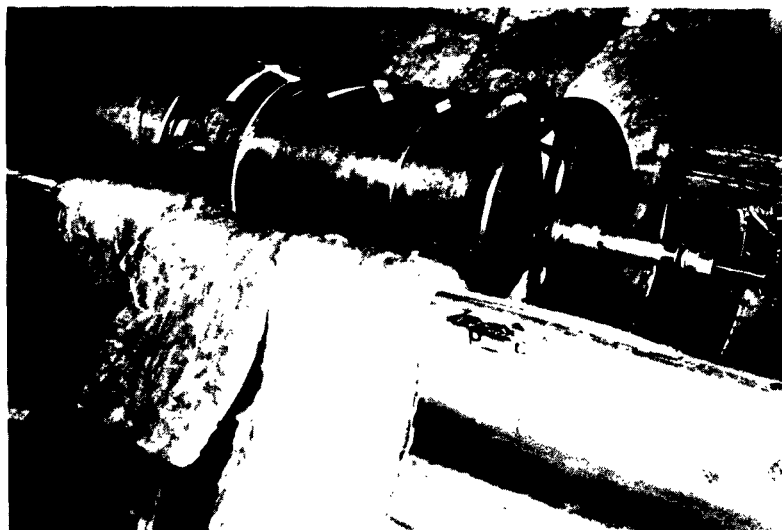


Figure 12. An Assembled and Thermocoupled Case Ready for Curing.



Figure 13. The Finished Components Used
in Prototype Bonded Joint Case Fabrication

In none of these first five tests was a catastrophic bonded joint failure effected. Instead some difficulty with other parts of the system was encountered. In tests one and two failure of the heavy marmon type clamp rings which secured the dome hydrotest heads to the bonded end rings occurred. In tests three and five rupture of the case wall occurred. Ordinarily it would be desirable in production cases to have the bonded joint stronger than the tubular section to assure full utilization of the case material. In this program however, it was more advantageous to effect failure of the bonded joint to permit direct examination of the adhesive failure pattern. In addition, through the appearance of the bond line, a more complete evaluation was possible of the effect of bonding variables introduced into the fabrication of each group of cases. In test number four a high pressure hydrostatic fluid leak developed through the bonded joint and the test had to be terminated.

The first problem, that of clamp ring failure, was solved by re-design and fabrication of heavier clamp rings. It was believed that as machining and bonding technique improved the joint leak problem would be solved, or eliminated, by the adhesive itself which would act as a sealant. Slightly increased adhesive flow from between the joints and more uniform bonding pressure would result in an even bead of adhesive covering and sealing the exposed joint end inside the case. As an added precaution however, Silastic RTV 731, a silicone rubber adhesive and sealant, was used to line the inner joint on all future cases.

It was evident that to solve the remaining problem, assuring failure of the bonded joint rather than the case wall, the bonded joint would have to be weakened. This was done to all succeeding cases by reducing the scarf joint length from 4" to approximately 2-1/4". The original goal of the program was to fabricate bonded joints having shear strengths of 3750 psi over each inch of the 4" long joint for a total strength of 15,000 pounds per peripheral inch. With the joint length reduction from 4" to 2-1/4" it would not be possible to achieve the latter figure, however the former figure, 3750 psi, could still be achieved regardless of joint length. It was reasoned that if 3750 psi shear strength could be attained over a joint length of 2-1/4" then, with a stronger and/or less notch sensitive case material, it could also be attained over a joint length of 4". This would be basically true only if bonded joint strength were directly proportional to joint length within the 2" to 4" range. Reference is made to Figure 14. This graph of joint strength versus joint length for FM-1000 adhesive was published by IKD in Quarterly Report Number 5, dated 15 January 1962 for Feltman Research and Engineering Laboratories, Picatinny Arsenal, Dover, New Jersey under Contract DA-20-018-ORD-22890. It shows that the function between these two variables in the 2 to 4" range is a straight line. The inference then was that if the unit shear strength of 3750 psi could be achieved on the shorter joint it would at least indicate that the original target strength of 15,000 pounds per peripheral inch could also be achieved.

Each point represents the mean value of five samples. Uniaxial lap shear samples were one inch wide with various lap lengths. The load corresponds to the longitudinal load per inch of periphery in a cylinder. The slope of the curve gives the unit lap shear stress.

Graph of Lap Shear Values

AF-15 ———— ⊙ ————
 FM-1000 ———— Δ ————
 XD-1078919 ———— ⊕ ————

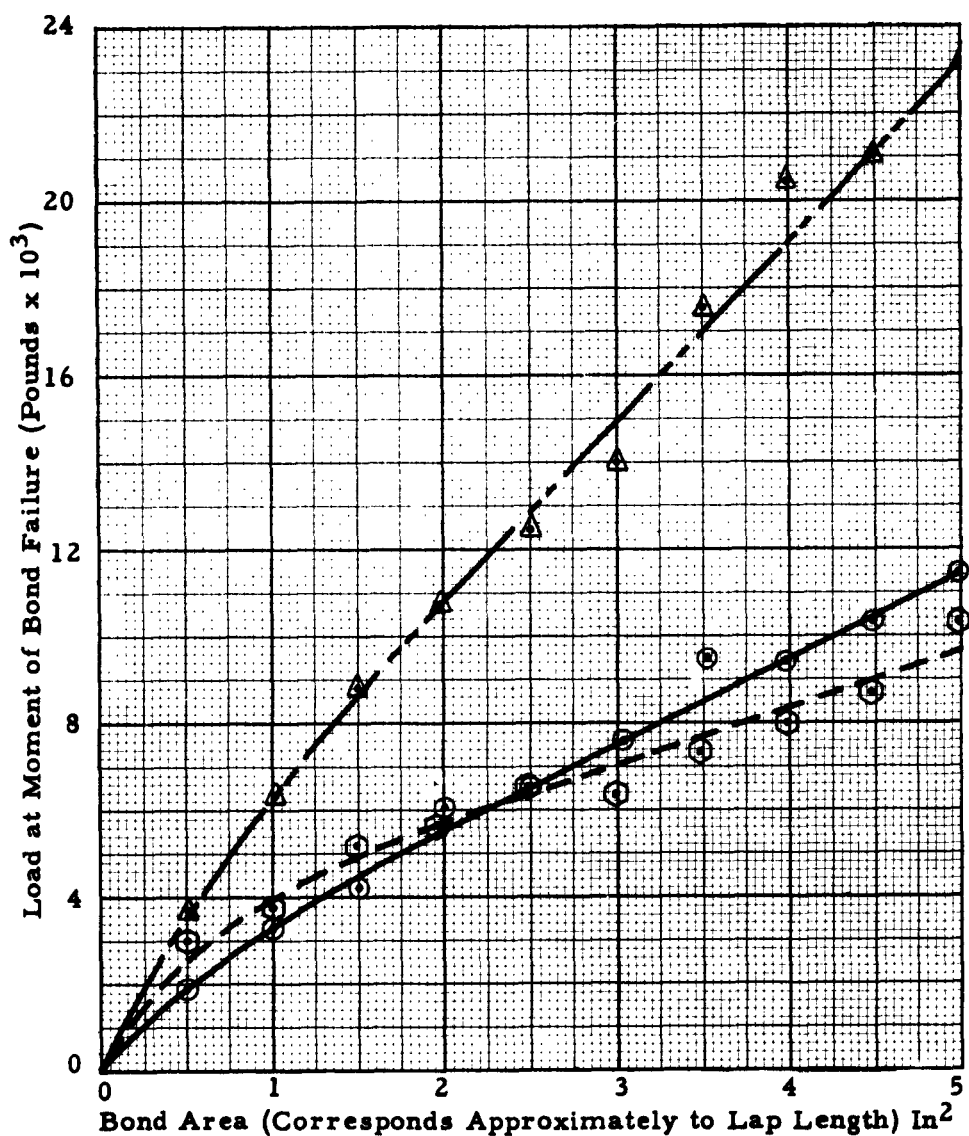


Figure 14. Load vs. Bonded Area for Various Lap Lengths

The ultimate bonded joint strengths of four of the five units could not be determined since these joints did not fail in hydrotest. The minimum strengths in terms of unit stress, of the first group of cases was as follows: #1 - 2550+ psi, #2 - 2440+ psi, #3 - 3420+ psi, #4 - 1390 psi, and #5 - 2900+ psi.

Photographs of cases #3 and #5 in which rupture of the case wall occurred prior to bonded joint failure are shown in Figures 15 and 16.

2. Group II Tests

The primary difference between the three cases in Group II and the five in Group I was that the former had the reduced scarf joint lengths. With the exception of slightly different curing temperatures on two of the units, all machining and bonding variables in Group II tests were held constant with respect to Group I.

Hydrotest of the three Group II cases produced the following joint strengths: #6 - 2930 psi, #7 - 2470 psi, #8 - 3750 psi. The target joint strength of 3750 psi was attained with unit #8. For this last case, hydrotest pressure, 1920 psig, was held for one minute. At 1600 psig on the second cycle joint failure occurred. Examination of the failed joints in cases 6 and 7 revealed that both shared a common joint gap defect which, from the appearance of the bond line, extended 4" - 5" peripherally and approximately $\frac{2}{3}$ the length of the joint. There was no doubt that these gaps between the mating scarf joint surfaces, present during the cure cycle, seriously weakened the joints.

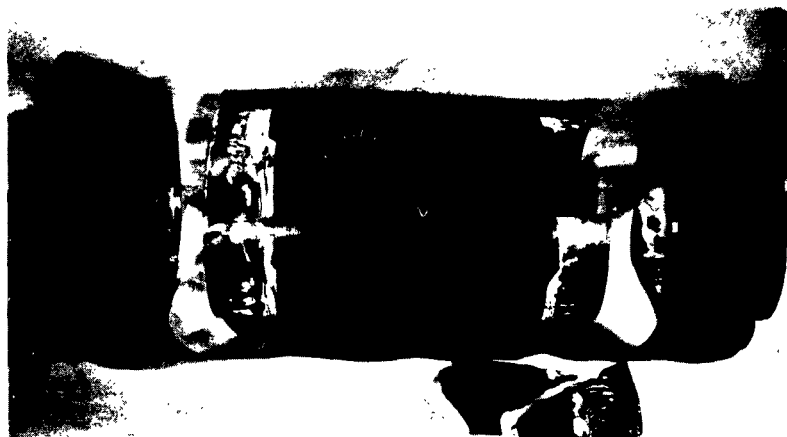


Figure 15. Prototype Case #3 Showing Where Rupture of the Case Wall Occurred. Explosive shock separated the bonded end rings from the tubular section.

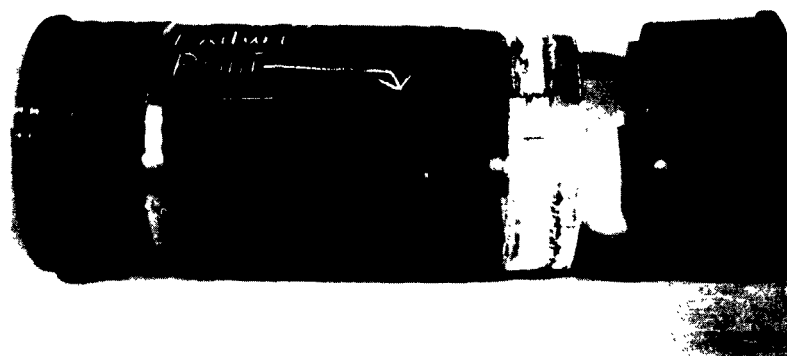


Figure 16. Prototype Case #5 Showing Where Rupture of the Case Wall Occurred. Explosive shock separated one end ring from the tubular section.

A minimum of 25 psi bonding pressure is required to obtain high strength bonds with FM-1000 adhesive. This requirement is, of course, not fulfilled where gaps exist between the surfaces to be bonded. It was evident that before higher joint strengths could be realized, joint gaps would have to be entirely eliminated.

The method of applying a longitudinal compressive force to force the scarfed surfaces into close contact thereby applying bonding pressure and eliminating joint gaps worked quite satisfactorily for the sub-scale cases. This was the method brought forth for prototype case fabrication. The prototype cases however were more out-of-round due to the spinning and heat treat operations than were the sub-scale cases which were machined from heavy walled aircraft quality seamless steel tubing. Due to the degree of component (end rings and tubular section) out-of-roundness and stiffness of the components, this longitudinal force method of gap elimination was not entirely effective for the larger cases. In the forthcoming Group III tests another approach to the gap problem would be tried.

The results of the eight bonded joint prototype case tests are shown in Table II.

III. Super Tricent Seamless and Welded Cases

The Super Tricent steel seamless and welded cases were incorporated in the program as a supplement to the work that was done with the MBMC #1 steel welded cases in which controlled surface decarburization was employed to reduce notch sensitivity.

Table II. Prototype Bonded End Closure Case Results

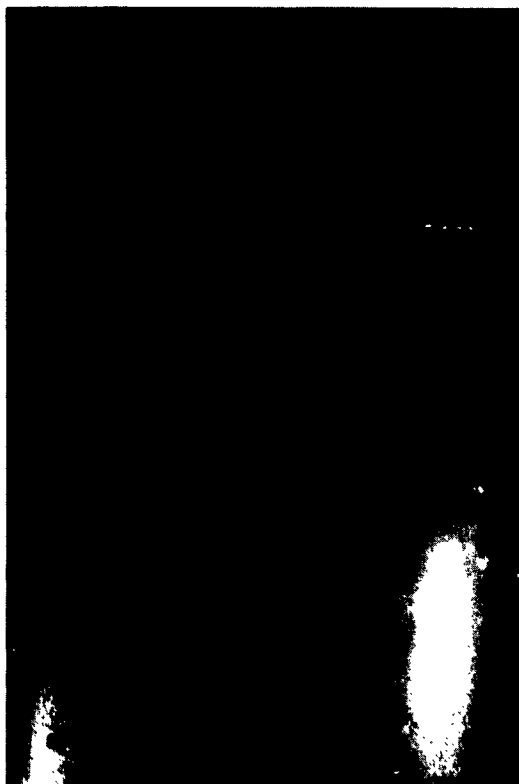
Test Number	Bonded Joint Length - inches	Joint Strength - psi
1	3-1/2	2550+
2	3-1/2	2440+
3	3-1/2	3420+
4	4.0	1390
5	4.0	2900+
6	2-3/32	2930
7	2-5/8	2470
8	2-3/32	3750

Note: Joint strengths shown for cases 1, 2, 3 and 5 are minimum values. Their ultimate strength could not be determined since these joints did not fail during hydrotest.

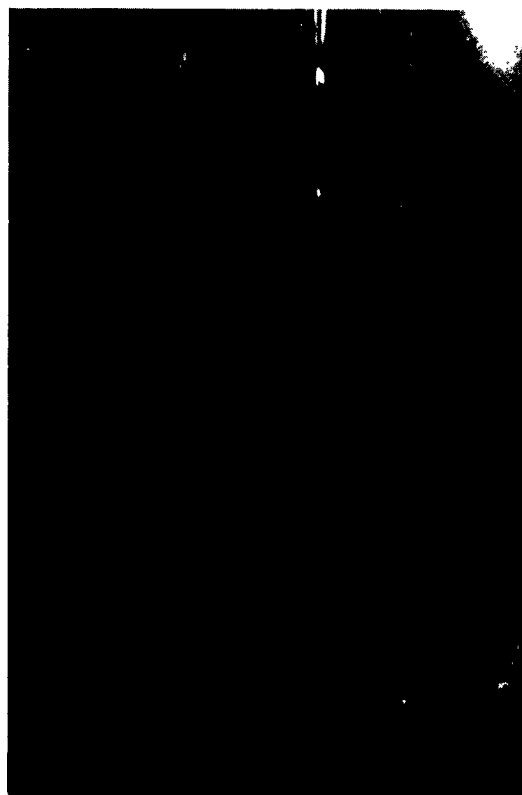
The results of this supplementary work were for the most part unsatisfactory. In the second quarterly report results of the first three of five seamless cases were discussed. Premature failure of the case wall occurred in each of these cases. Cases #4 and #5, fabricated during this reporting period, were re-heat treated to produce a greater depth of decarburization in an effort to prevent premature failure. Case #4 suffered premature failure. Case #5 was successful in that it reached a hoop stress of 283,000 psi.

Four Super Tricent steel welded cases were fabricated during this period. Only one of the four cases reached its full potential strength. Premature rupture of the case wall occurred during the other three hydro-tests. As was done with the seamless cases, the welded cases were strain gauged and stresscoated in an effort to locate possible stress concentrations. Excellent gauge readings and stresscoat crack patterns were obtained, however, again as with the seamless cases, no severe stress concentrations were found. The stresscoat was applied to the welded case at the junction of the longitudinal and girth welds, the most likely areas of stress concentration. Photographs of the crack pattern are shown in Figures 17 and 18.

Since this supplementary work represented only a small portion of the over-all project and was done under a relatively low level of effort no intensive metallurgical investigation was undertaken to determine the exact cause of these premature failures. It was believed however that the quality of the Super Tricent air melt steel combined with possible over-spinning and



Figures 17 and 18. Stresscoat applied to the areas of most likely stress concentration, at the junction of the longitudinal and girth welds. Note the relatively uniform crack pattern indicating negligible stress concentrations.



corresponding severe cold reduction, particularly on the last pass, may have been responsible for premature failure. It has been IKD's experience that over-spinning results in multiple minute crack formations occurring on the inside of the case wall. This theory of over-spinning is supported somewhat by the appearance of the failure initiation areas in four of the five seamless cases shown in Figures 19 through 22. These failure initiation areas appear to have originated from the inside of the case wall. A table of power spinning data for the Super Tricent steel seamless and welded cases is shown in Table III. This spinning procedure and per cent reductions for each pass was similar to that used for the MBMC #1 welded cases all of which burst at high strength levels. The difference may have been that due to the higher carbon content the Super Tricent steel would not tolerate the same amount of cold reduction as did the lower carbon MBMC #1 steel.

The results of the Super Tricent seamless and welded cases are shown in Tables IV and V.

FUTURE PLANS

During the next quarter it is expected to (1) complete the fabrication, testing and evaluation of the prototype bonded end closure cases and (2) write and issue the final report.



Figure 19. Super Tricent Case #2
Plain stress area originates from
a crack at the inner wall surface.



Figure 20. Super Tricent Case #3
Plain stress originates from a
crack at the inner wall surface

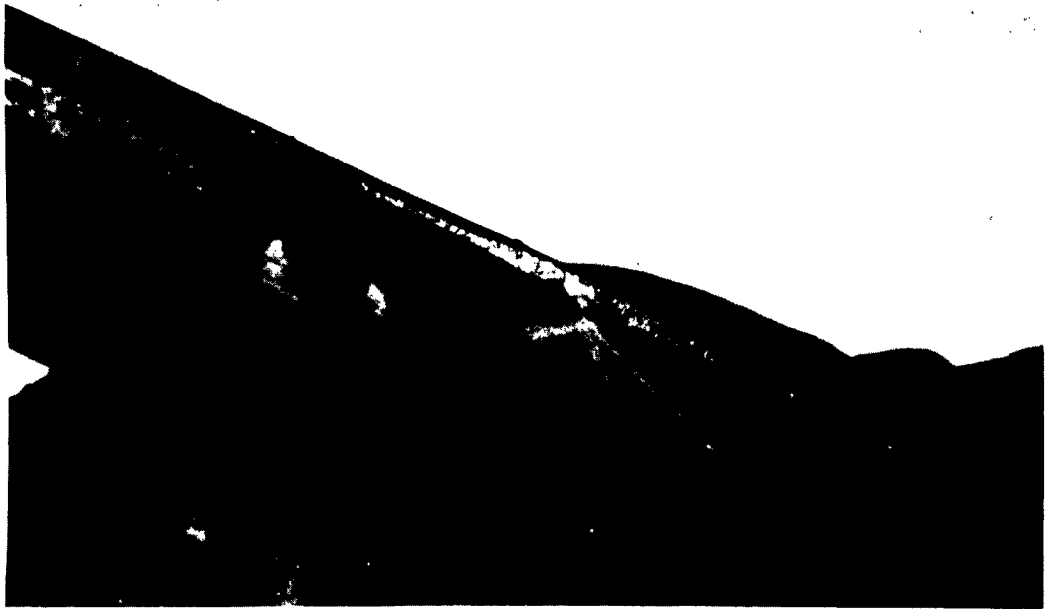


Figure 21. Super Tricent Case #4
Plain stress area originates from
a crack at the inner wall surface.



Figure 22. Super Tricent Case #5
Plain stress area originates from
a crack at the inner wall surface.

Table III. Power Spinning Data for Super Tricent Steel Seamless and Welded Cases

Feed: 9" per minute

RPM: 250

<u>Starting Wall Thickness</u>	<u>1st Pass</u>	<u>2nd Pass</u>	<u>3rd Pass</u>	<u>4th Pass</u>	<u>5th Pass</u>
.550"	.450"	.325"	.195"	.110"	.060"
<u>% Reduction</u>					
0	18	28	40	44	45

Note: All cases were stress relieved after the 3rd, 4th and 5th passes.

Table IV. Super Tricent Seamless Case Results

Case No.	Depth of Surface Decarburization Inches	Inside Diameter	Wall Thickness Inches	Hydrotest Pressure psig	Hoop Stress psi x 10 ³
1	.003-.004	16.300	.058	1825	256.2
2	.005	16.296	.055	1150	170.0
3	.005	16.308	.057	960	137.5
4	.007	16.447	.058	1350	191.5
5	.007	16.265	.051	1775	283.0

Table V. Super Tricent Welded Case Results

Case No.	Depth of Surface Decarburization Inches	Inside Diameter	Wall Thickness Inches	Hydrotest Pressure psig	Hoop Stress psi x 10 ³
1	.008-.009	16.264	.062	1600	210.0
2	.008	16.235	.057	2010	287.0
3	.007	16.225	.059	1320	181.5
4	.007	16.185	.060	1400	188.8

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