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[4	METASTABLE AUSTENITIC FORMING OF HIGH STRENGTH PRESSURE VESSELS
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33 531 300 Lycoming Division - AVCO Corporation Stratford, Connecticut Metastable Austenitic Forming of High Strength Pressure Vessels 9) Semiannea (Interim Technical Progress Repert. no 2) 1 Sep 62 - 30 Mar 63, Prepared for NĤ Aeronautical Systems Division Wright Patterson Air Force Base U 15 Contract AF33(657)-7955 Ap**ril (**15 ₿63 NH NA 24 Prepared by Approved by: M. Rayn W. R. Freeman, Chief, Manager, Materials Engineering Materials Laboratory Concurred by: Approved by: Mihalek Project Engineer Superintendent, Manufacturing Eng.

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FORWARD

This Interim Technical Documentary Progress Report covers the work performed under Contract AF33(657)-7955 from 1 September 1962 to 30 March 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with the Lycoming Division, AVCO Corporation, Stratford, Connecticut was initiated under ASD Project No. 7-887, "Metastable Austenitic Forming of High Strength Pressure Vessels". It is being accomplished under the technical direction of Mr. J. O. Snyder, Manufacturing Methods Branch, Manufacturing Technology Laboratory, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Messrs. J. M. Raymer, Chief of Materials Engineering, Materials Laboratory and F. Mihalek, Chief Process Engineer were the engineers in charge of this project. Others who co-operated in the research and in the preparation of this report were: August Alexander, Senior Development Engineer, Dr. H. Klein, Chief of Mechanics and Dynamics and Experimental Stress Analysis, Joseph Fekete, Group Leader, Process Engineering, and John Erinakis, Process Engineer.

The primary objective of the Air Force Manufacturing Methods Program is to develop a high performance integral rocket motor case from metallic materials with improved mechanical and design properties. This program encompasses the utilization of the shear spinning process for motor case fabrication and the evaluation of the deformation of steels while in the metastable austenitic condition as a means of enhancing its performance.

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional development required on this, or other subjects, will be appreciated.

ABSTRACT

During the second semiannual project period, Phase I, of the Contract No. AF33(657)-7955 essentially was completed. Three selected alloys; Type H-11 tool steel, AM 355 semiaustenitic stainless steel, and 18NiCoMo (300) maraging steel, were fabricated into biaxial pressure vessel test specimens. For the fabrication of the biaxial pressure vessel test specimens, designed experiments were utilized to evaluate a variety of processing and heat treat variables. The fabricated pressure vessels (i. e. tubes) were tested to failure in a hydrostatic test facility and evaluated for selection of an optimum material and associated fabrication process for a high performance, integral rocket motor case. Based on these studies the 18NiCoMo (300) maraging steel and a specific processing schedule were selected for Phase II and III evaluation. An intermediate size cylindrical test specimen and an integral subscale rocket motor case were designed for Phase II investigation of optimized fabrication techniques for the manufacture of an integral motor case from 18NiCoMo (300) material. The forgings and tooling for fabrication and testing in Phase II were ordered.

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INTRODUCTION

The present and future goals in missile and space vehicles emphasize the need for developing materials and/or material processes for producing large diameter thin wall missile and rocket motor cases with improved mechanical and design properties. Recent investigations have shown that the strength levels of certain steels may be increased by deformation while the material is in the metastable austenitic condition. However, the methods of deformation (i. e. stretching, rolling, forging) used in evaluating such material behavior currently are not applicable for the production of large diameter, thin wall integral rocket motor cases. The shear spinning \smile process is used extensively at Lycoming to produce cylindrical, conical, parabolic, and a variety of other geometric forms from a variety of materials and offers a deformation process with such capabilities.

The purpose of this investigation is to select a material and to develop a process for fabricating an integral rocket motor case (with no weldments) by the shear spinning process and to evaluate the deformation of the material while in the metastable austenitic condition as a means of achieving a motor case that will develop hoop strength values in excess of 300,000 psi.

This is the second semiannual interim technical report issued under Contract AF33(657)-7955 and it summarizes the experimental work conducted in Phase I on the Type H-11 hot work steel, AM 355 semi-austenitic steel, and 18NiCoMo (300) maraging steel during the period ending March 31, 1963. This experimental work in Phase I has resulted in the selection of a material and specific shear spinning parameters for Phase II effort involving fabrication of subscale integral rocket motor cases.

The first semiannual progress report reviewed the literature of the materials potentially suitable for fabricating an integral rocket motor case by the shear spinning process. Three materials were selected as representative of different categories of high strength steels and procured for experimental evaluation in Phase I of this program by the fabrication and biaxial testing of subscale pressure vessels.

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DISCUSSION

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1. Current Program Status:

During the second semiannual reporting period, all of the Phase I subscale pressure vessel test specimens of H-11 tool steel, AM 355 semiaustenitic stainless steel and 18NiCoMo (300) maraging steel were fabricated by shear spinning and machined into biaxial test specimens. During the fabrication of these test specimens the many processing variables to be investigated were evaluated using a statistically designed experiment. The initial series of tests, consisting of a fractional factorial experiment using the Hyper-Graeco-Latin Square with five variables at each of four levels was completed and evaluated on all three materials. On the basis of data from the initial tests, 18NiCoMo (300) was selected as the material to be used in the Phase II and Phase III sections of this contract. A full factorial experiment was then designed and completed with the 18NiCoMo (300) steel to further evaluate and define processing variables.

An intermediate size subscale cylindrical pressure vessel, closures and an integral subscale rocket motor case and closure were designed for Phase II. The 18NiCoMo (300) maraging steel forgings for Phase II have been ordered, and additionally, the spinning mandrel, rollers, and closures for testing the cylindrical pressure vessel have been ordered.

2. Shear Spinning:

The first semiannual report discussed in considerable detail the shear spinning process, the selection and approach for evaluation of numerous materials, the processing variables and their subsequent effect on product properties, and the effect of both material characteristics and processing variables on shear spinning fabricability, therefore, these topics will not be reiterated in this report. Tables I, II, and III present the variables evaluated for the three selected alloy steels of H-11, AM 355, and 18NiCoMo (300) in the initial fractional factorial experiment. All of these tests were performed on single heats of each material supplied in accordance with existing specifications. Chemical analyses and mechanical property capability tests are presented in Table IV.

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Austenitizing, or solutioning, of all shear spin preform blanks were accomplished in an inert atmosphere of argon to prevent excessive decarburization and high temperature oxidation. Decarburization is undesirable since it would prevent obtaining maximum uniform hardness throughout the spun specimen. Both decarburization and oxidation are not considered as detrimental to the fabrication process if the degree is not excessive. After holding at temperature for the prescribed length of time the blanks were transferred to the preheated spinning mandrel and equalized at the spinning temperature to be evaluated. A single, or multiple, "back extrusion" pass was then performed at the required total reduction and the shear spun cylindrical test specimen removed and air cooled to room temperature. They were subsequently subzero cooled to 100° F. for 3 hours in a deep freeze chamber, and then tempered in an air atmosphere furnace at the required temperature and time.

The specific shear spinning and processing history for each material fabricated in the initial statistical experiment was as follows:

Type H-11 Tool Steel -

The initial specimens were reduced 25%, equivalent of a roller bite of 0.060", with a single pass at a starting temperature of 600° F., however, during the spinning there occurred temperature rises of 160°F. in the material extruding from under the rollers. Figure 1 presents a typical reproduction of the actual spinning temperatures recorded and shows the rapid temperature rise with even a low percentage reduction. Such uncontrolled, or excessive temperature rise during deformation is undesirable since in the shear spinning of a high strength martensitic type material the temperature must be maintained within the austenitic bay of a T-T-T curve. Any significant temperature increase during spinnings causes formation of undesirable non-martensitic products or effects a recrystallization of the deformed material which results in either no significant improvement or an actual decrease in yield and ultimate strength of the finished spinning. Reduction in temperature change can be obtained by several means whereby more time was available for the dissipation of a given amount of heat generated by deformation; these variables include increasing mandrel RPM, decreasing roller feed or percent reduction, and/or providing external cooling. In the fabrication of the biaxial test specimens internal heating was effectively controlled by varying the

mandrel speed and roller feeds. The remaining test specimens of 25% reduction were satisfactorily shear spun incorporating the variables as defined in Table I.

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Initial attempts at obtaining 50 percent (roller bite of 0.375") reductions with a single pass were unsuccessful as a result of a large volume of metal being worked and extruded with considerable heat being generated. The heat not having time to diffuse created a steep temperature gradient at the point of roller contact with maximum temperatures of 1500° F. being observed ahead of the roller. These large temperature increases at the roller surface, in addition to the normal temperature effects on structure, also caused steep thermal gradients through the material cross section being extruded back under the rollers and resulted in improper extrusion and buildup of material ahead of the rollers which prevented the spinning of a complete part. To correct this problem, the preform blanks were modified by machining prior to the 50 percent reduction pass (roller bite reduced to 0. 190") and the test specimens spun successfully. Initial temperature rises of 500°F. were encountered, however, this was reduced to 200°F. by increasing mandrel RPM and decreasing roller feed.

Single pass reductions of 75 percent (roller bite of 0. 560") were unsuccessful because of excessive overheating and failure to extrude properly. Attempts to spin the 75 percent reduction (total) with two passes (roller bite of 0.280" on each pass) were also unsuccessful. A preform blank was modified to a wall thickness of 0. 570" and shear spun (using a roller bite of only 0.207") with two passes. However, spinning an identical preform blank at lower temperatures proved unsuccessful. The parts spun successfully on the first pass but failure continually occurred on the second pass. Many attempts were made to adjust mandrel speed and roller feeds however a successful processing technique could not be developed to produce a 75% reduction in either a single or multiple pass. The lack of success in achieving high reductions for H-ll is not too surprising since the temperature rise from the internal generation of heat during reduction(s) is intentionally minimized by means and for reasons already presented thereby inducing martensite transformation and preventing relief of internal stresses. Since thermal stress relieving operations cannot be utilized between passes there exists a definite limitation in shear spinning high total reductions while maintaining the material within the austenitic bay of the T-T-T curve.

Table V summarizes the detail processing conditions utilized in the fabrication of the H-ll biaxial test specimens.

AM 355 Semi-Austenitic Stainless Steel -

The first AM 355 specimens were reduced 25% (roller bite of 0.060") with a single pass at room temperature, however, during spinning there were temperature increases of up to 150° F. in the material being extruded behind the rollers and the cylinder developed axial and circumferential cracks in the last one inch of spun section. Additional attempts to spin the AM 355 at room temperature when solutioned at $1710^{\circ}F$. in accordance with the statistical experiment were not successful. When solution treated at 1710°F., the Ms of this alloy is above room temperature and although the transformation to martensite is only partial, the spinning capability is seriously affected as the deformation temperature decreases. This alloy also becomes quite sensitive to strain induced transformation to martensite as the prior solution (conditioning) temperature decreases which further decreases formability. The 1710°F. treatment, which is also a conditioning treatment for the alloy produces a slight carbide precipitation in the grain boundaries which may adversely affect deformation behavior. The specimens solution treated at 1900°F., 1600°F., and 1375°F. were shear spun successfully to 25% reductions at 160°F., 300°F., and 450°F., respectively. In each of these latter processing histories the solution and/or spinning temperatures used were such to result in the spinning being performed while the steel was fully austenitic.

Single pass reductions of 50 percent (roller bite of 0. 190") were produced successfully in only two of the four statistical combinations to be evaluated. With the greater roller bite required to produce a 50% reduction, strain induced transformation of austenite to martensite was increased with the expected resultant decrease in ductility and formability of the material. The increased reductions attempted therefore make the selection of solution and spinning temperatures even more restrictive if the alloy is to remain austenitic. In this respect the high solutioning temperature of 1900°F. yielded the most stable structure which could be shear spun 50% successfully. This particular test specimen also exhibited substantial internal heating (approx. 500° F.) which undoubtedly aided in the deformation of non-martensitic material. Attempts to make 50% reductions at 450° F. were unsuccessful, however, a low solution temperature of 1375°F. was used which resulted in substantial carbide precipitation thereby raising the Ms to its highest temperature. A test specimen (#24) solutioned at

1600°F. was successfully shear spun at 300°F.

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No single pass reductions of 75 percent were successfully completed because the volume of material being flowed under the rollers was so large and the force required to do this caused pieces to flare at one end. Attempts to achieve 75% total reduction in two passes were also unsuccessful. Specimen number 51 was solution treated at 1600°F., air cooled, and shear spun at room temperature. The part broke into two pieces after spinning a half inch of reduced section during the first pass. Specimen number 27 was solution treated at 1375°F., air cooled to 150° F., and shear spun successfully on the first pass but developed severe cracks on the second pass. Specimen number 19 was solution treated at 1710°F., air cooled to 450°F., and spun at 450°F. The part developed surface cracks during spinning and also flared on one end. Specimen number 52 was solution treated at 1900°F., air cooled to 300°F. and shear spun immediately; this part also developed severe surface cracks and the end of the part against the stripping ring flared. Specimen number 73 was solution treated at 1710° F., air cooled to 450° F., and spun at that temperature. The part was deformed successfully on the first pass but cracked severely on the second pass.

Table VI summarizes the processing conditions utilized in the fabrication of the AM 355 biaxial test specimens.

18NiCoMo (300) Maraging Steel -

The shear spinning of 25% reductions was successfully performed on the 18NiCoMo (300) without any appreciable difficulty. As in the case of the other materials the problem of internal heat generation was again encountered, however, the adjustment of machine variables effectively minimized it to an average of 150° F. It was observed that the higher spinning temperatures tended to result in increased metal buildup due to the formability of the alloy, however this did not present too serious of a problem and can be rectified by roller design.

Single pass reductions of 50 percent were made successfully on test specimens shear spun at room temperature, 200°F., 400°F., and 500°F. The specimen temperature during spinning increased approximately 600°F. on some test specimens, however by an appropriate adjustment of roller feed and RPM, the temperature increase during spinning was reduced to 100°F.

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Initially, a single pass 75 percent reduction was attempted with a roller bite of 0.55", however, the equipment did not have the capacity to extrude this amount of material. Shear form blanks were then remachined to a starting blank thickness of 0.555" for shear spinning attempts in two passes (0.207" bite) at room temperature. Test specimen 53 was solution annealed at 1600° F., air cooled to 500° F., and shear spun successfully in two passes. Utilizing this modification of the shear form blank the material was then successfully shear spun at room temperature in two passes. Subsequent to this, a series of test samples were shear spun at both 200° F. and 400° F. in accordance with the statistical experiment, however no dimensionally acceptable test specimens could be made. Attempts to spin at these two temperatures produced various fabricability behaviors (e. g. roller buildup, bell mouthing, etc.). It is felt that these problems could have been solved, however lack of available material prevented this.

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Table VII summarizes the processing conditions utilized in the fabrication of the 18NiCoMo biaxial test specimens.

On completion of all the shear spinning of biaxial test specimens and the hydrostatic testing of these specimens, a review was made to select a single material that showed the greatest potential for achieving an integral rocket motor case that would develop hoop strength values in excess of 300,000 psi. For reasons to be explained later, the 18NiCoMo (300) maraging steel was selected for continued study to achieve the contractural goals of the program. The second step of the shear spinning effort was then programmed to better define the processing variables which showed greatest significant effect in the shear spinning of this steel. As in the first evaluation a statistical approach was again used to minimize the number of specimens and to evaluate the interactions of combined variables. A full factorial experiment using four variables at each of two levels was utilized. Table VIII shows the variables and constants included for each test specimen (total of 16) which were selected from the results of the fractional factorial experiment. Those variables of special interest are deformation temperature (R.T., 500°F.), reduction (40, 65%), number of passes (1, 2), and aging time (3, 6 hrs.). Constants are as follows: solution temperature (1500°F.), solution time (1 hr.), quench rate (air cool), subzero cool (-100°F. /3 hr.), and aging temperature (900°F.).

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With the experience gained in the shear spinning of the first statistical sampling group the fabrication of the second sampling group was accomplished without any difficulty. The overall spinning behavior of the 18NiCoMo (300) for all selected processing conditions was rated as very good. In the spinning of biaxial test specimens at 500° F. the temperature rise due to internal heating was limited to a maximum of 60° F. For room temperature spinning where more machine power was required, the temperature rise approached a maximum of 300° F. However, it is felt that some temperature increase at the rollers due to heat of deformation is beneficial to the extrusion of the material under the roller as long as detrimental metallurgical effects do not result. Of course, excessive temperature increases result in steep thermal gradients which adversely affect spinning behavior.

Table IX summarizes the processing conditions utilized in the fabrication of the second statistical sample of the 18NiCoMo biaxial test specimens.

3. Biaxial Pressure Vessel Testing:

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Subsequent to the shear spinning and heat treating of biaxial test specimens in accordance with the processing conditions of the statistical experiment they were machined to the dimensional requirements as shown in Figure 2. The biaxial test specimens were then instrumented with six strain gages (Budd Metalfilm Type C6-141) positioned at critical strain areas to provide axial and circumferential strain measurements every 120°. The biaxial test specimen design and critical strain areas had been previously verified by stress-coat analyses. After strain gaging, the test specimens were assembled into the Laboratory hydrostatic test stand shown in Figure 3 and pressurized to failure. Pressure and strain were automatically recorded during test.

After failure of a biaxial test specimen, the automatically recorded pressure and strain data was used to determine nominal ultimate strength, nominal yield strength (0.2% offset) and total gross strain. Nominal stresses were computed from internal pressure measurements and original dimensions. Strain was obtained from the temperature compensated strain gages. No corrections were made for bulging of the vessels since such corrections were relatively minor considering the large scatter in results due to processing factors. Total gross strain

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was determined from dimensional measurements of the specimen before and after test. In some instances the offset yield strengths were not determinable due to the fracture occurring intermediate to two strain gage groupings on the test specimens and to catastrophic failure at extremely low total strain; for similar reasons the total gross strain could not be ascertained in these cases.

Biaxial test specimens were sectioned for fracture analysis and determination of point of origin wherever possible. Hardness checks were made at the point of origin and a microexamination performed along the full gage length to determine structural characteristics. Presented in this interim report is primarily the mechanical property data obtained from the biaxial tests. A more detailed analysis of microstructural characteristics and property correlation with processing history is underway and results will be presented in the next interim report.

The specific observations in the biaxial testing for each material fabricated was as follows:

Type H-11 Tool Steel -

A tabular summary of test results on H-11 burst specimens is included in Table V and representative stress-strain curves are shown in Figure 4. A maximum hoop strength of 413,000 psi was obtained on the H-11 test specimen which had been deformed at 1000° F. using 50% reduction in one pass and subsequently double tempered at 700° F. The failure was a typical cleavage type fracture with a very small shear lip. This specimen also exhibited the highest determined yield strength of 392,000 psi at a hardness of Rc 62. Minimum strength of the statistical samples was 233,000 psi which was obtained with a tube that had been deformed to a 25 percent reduction, however, there is some question as to the validity of that particular test result.

Examination of the fracture origins on all specimens tested indicated that the fractures primarily originated at the O. D. and near the center of the gage length as predicted by initial stress analysis. Failure in sample numbers 1, 3, 9, 10, 12, 18, and 66 occurred with an area of slow initial crack growth which developed into rapid crack propagation by ductile shear failure. However, failure in sample numbers 13, 14, 16, 17, and 65

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occurred with an initial area of slow crack growth which developed into rapid cleavage mode of failure with little plastic flow occurring. Figures 5 and 6 depict several of the test specimens which are typical for H-11 that failed by a ductile mode. The biaxial test specimens remained in essentially one piece when the failure was of the ductile type, although occasionally several small fragments were separated from the major section. Figures 7 and 8 depict several of the test specimens exhibiting brittle fracture behavior and a typical cleavage failure. The pressure vessels failing in a brittle manner broke into numerous fragments as is also shown in the aforementioned figures.

Review of the statistical program test data indicates that certain of the processing variables produce significant trends in tensile properties whereas others have little effect. Recognizing the limitations of the sampling approach as to the relative importance of the interacting variables, it may be said that the highest burst strength is obtained with: (1) a solution temperature of 1875° F. (with a more rapid dropoff in properties with decreasing temperatures), (2) a tempering temperature of 700° F., and (3) increasing percent spinning reductions. A spinning temperature of 900° F. apparently produces a minimum rather than a maximum in properties in that high strengths were attained when deformation occurred at both 600° F. and 1000° F. Prior solution treatment times has little effect on resultant properties within the time period investigated.

AM 355 Semi-Austenitic Stainless Steel -

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A tabular summary of the AM 355 test specimen results is shown in Table VI and representative stress-strain curves are presented in Figure 9. AM 355 austenitic stainless steel biaxial test specimens exhibited the lowest average hoop strength with only one specimen having a hoop strength in excess of 300,000 psi. The AM 355 material had the lowest hardness and the highest percentage elongation. Considerable plastic deformation occurred in the AM 355 test specimens as shown by the bulging depicted in Figure 10. The failure in all of the AM 355 specimens initiated with an area of slow crack growth which developed into a rapid failure by ductile shear mode. Most AM 355 test samples exhibited some degree of tensile instability in that there was considerable plastic flow to rupture with no increase in load. The levelling off of the stress-strain curves tend to indicate this behavior. Figures 11 and 12 depict several typical failures as observed in AM 355 test

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specimens. Due to the lack of test data available and the unacceptable strength levels developed, no detail analysis of processing variable effects was attempted on this alloy.

18NiCoMo (300) Maraging Steel -

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In the first statistical sample of specimens evaluated for the selection of a material for Phases II and III of this program, 14 biaxial pressure vessel specimens were tested to failure and the results are included in Table VII. Representative stress-strain curves are presented in Figure 13. This first series of 18NiCoMo (300) maraging steel specimens had ultimate hoop strength values ranging from 236,000 to 368,000 psi with correspondingly high yield strengths and reasonable values of total gross strain.

The failure mode in all of the test specimens was by a ductile shear mode with no evidence of brittle fracture even at the highest strength levels. Failures occurred in most cases after a considerable amount of plastic flow. The failure origin was very difficult to locate and was not clearly defined by an area of flat fracture and was usually located by a slight change in texture of the fractured surface or the change in direction of shear planes. Figure 14 are photographs depicting typical failure mode and fracture characteristics.

Analysis of the first statistical program indicated certain trends of properties versus processing variables. In reviewing the data, one test result (Sample Nr. 45) was disregarded as questionable; this test will be repeated as time permits within the next reporting period. For maximum burst strength the optimum degree of reduction is at 50% and the spinning temperature can be either at room temperature or 500° F., the worst results being produced at the two intermediate temperatures. In neglecting the aforementioned questionable test result, it would appear that prior solution time and temperature are relatively unimportant, however, final resolution awaits further tests. Tempering temperature exerts a strong influence on ultimate properties with the optimum being at 900° F. By decreasing the tempering temperature, properties drop of sharply.

The above observations were employed in establishing the parameters for the following full factorial experiment which is detailed in Table VIII. Variables determined as of predominant significance and investigated were: (1) percent reduction, (2) deformation temperature, (3) number of passes, and (4) tempering time.

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Representative stress-strain curves for the second statistical sample are shown in Figure 15. Reductions both below (40%) and above (65%) the initial 50% "optimum" indicated increasing strengths with the higher reduction as shown in Table IX, however, it must be recognized that an optimum could exist between these two points. Results (Table IX) indicate that a deformation temperature of 500° F. is slightly more beneficial than room temperature, although the improvement is small (5,000 psi). Table IX also indicates that the longer aging time (6 hr.) decreases burst properties significantly and that reduction in two passes appears superior to one. The latter observation may be incorrect since two of the test specimens (Nrs. 69, 71) reduced in two passes had internal defects and were not included in the trend analysis. The degree of improvement ranged from only 5,000 - 10,000 psi for each variable, however, most improvements can be expected to be cumulative. From this, sample number 87 should have the optimum properties, but this is not the case. It differs in processing from the highest strength burst tube only in the number of passes required for total reduction. This is perhaps explainable by the defective test results mentioned above. Retest of specimens produced under the conditions employed for specimen numbers 69 and 71 should clarify the effect of number of passes and consequently the need for repeating the test conditions of specimen number 87.

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All of the samples had hoop strength values higher than 334,000 psi except the one which had a low value because of a known defect. The defect was a series of internal cracks which developed during the spinning operation and were not detected until subjected to hydrotest. Failure occurred after a considerable amount of plastic flow in almost all of the specimens. The mode of failure was by an area of slow crack propagation which developed into a rapid failure by ductile shear. None of the specimens tested exhibited brittle cleavage type failure. A reasonable degree of total strain (2-4%) was observed in most specimens. Figure 16 is a photograph of sample number 88 which had the highest hoop stress (383,000 psi) of this group of specimens. The failure origin was in the approximate center of the gage length.

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4. Selection of Material for Phase II and III:

The ultimate goal of this investigation is to produce an integral full scale rocket motor case approximately 120 inches long by 44 inch diameter fabricated by the shear spinning process and utilizing no welding. It is also the aim of this investigation to select the material to be utilized and to evaluate the effectiveness of shear spinning the material while in the metastable austenitic condition as a means of obtaining a fabricated case that would develop hoop strengths in excess of 300,000 psi. It is required that any rocket motor case so fabricated not exhibit a catastrophic failure behavior but show ductile fracture and exhibit good notch toughness.

The above listed objectives calls for a unique set of properties in a material. Based on these requirements, a group of three materials were selected for evaluation as presented in this report. These steels represented one steel from each of three different basic types showing potential of achieving all the requirements of the program. This selection represented a group of ultra-high-strength steels which obtain their properties through three different strengthening mechanisms and whose transformation characteristics are such as to make feasible the fabrication of an integral full scale rocket motor case.

In this investigation a statistical approach was utilized in an attempt to provide maximum data on processing variable effects and interactions, and establish an optimum combination of material, process, and strength characteristics as rapidly as possible. For this reason the actual shear spinning operation was used and strength properties in a biaxial stress field determined.

In the selection of one of the three steels for continued effort in Phase II and Phase III of this program, an equal degree of emphasis was placed upon: (1) the "spinnability" of the material, (2) the complexity of the full scale fabrication process of a rocket motor case, and (3), the mechanical properties and failure characteristics in a biaxial stress field. The final selection would then be based on an integration of these factors and not upon any single factor alone.

Page 14.

The shear spinning of H-11 tool steel yielded significant increases in strength by deforming while in the metastable austenitic condition. From this criteria, the attainment of 300,000 psi minimum burst strengths would not be a problem, however, in this respect the burst failures were predominantly characterized by extremely low plastic strain and brittle fracture behavior. It is probable that a more optimum combination of processing and heat treatment conditions could be established to reduce strength to a lower level with an attendant gain in ductile fracture behavior. If this could be successfully accomplished, the intended manufacturing process still remains extremely complex and impractical. Such a process would require continual fabrication of the motor case in the vicinity of 600°F. or 1000°F. + 50°F. and would require extensive temperature control and handling devices especially if the higher temperature were selected due to equipment power limitations (i. e. lower material strength at higher temperature). The use of multi-mandrel operations, which is a must in the fabrication of an integral motor case, would be complicated and costly. The tolerance for error in the manufacturing process would be extremely close, and since this is an irreversible process, a high scrap rate must be considered as a probability. It is therefore important that all these factors be considered prior to the selection of a material for Phase II and Phase III effort.

1

The fabrication and biaxial evaluation of the AM 355 semi-austenitic stainless steel did not make it appear as a probable selection. Most important, the AM 355 did not achieve the strengths required for a 300,000 psi minimum burst rocket motor case. The fabricability of the alloy was poor and considerable difficulty was encountered from strain induced transformation to martensite which produced immediate cracking under the rollers. Such occurrences would require frequent intermediate heat treatments and produce numerous problems in the shear spinning of a full scale case where large volumes of material have to be displaced for considerable distances on a mandrel.

In the case of the 18NiCoMo (300) maraging steel it was established that biaxial strengths in excess of 300,000 psi could be produced consistently. Strengths approaching that obtained in H-11 can be obtained which exhibited completely ductile failure behavior. It was also found that the 18NiCoMo showed the best "spinnability" of the three materials evaluated. Shear spinning at room temperature and 500°F. was excellent and produced strengths well above 300,000 psi. The capability of this material to be shear spun at either room temperature or 500°F. with excellent formability and to result in increased biaxial strengths in excess of the contractural requirement increases the chances of fabricating an integral rocket motor case significantly. It is entirely possible that the spinning of cylindrical sections could be performed at 500°F. for maximum strength, and the balance of the fabrication (e.g. shrinking of the aft end) be completed at either 500°F. or room temperature. The lower deformation temperatures coupled with room temperature fabricability provides a material with several practical advantages over the H-11 type steels, especially in the area of economics.

After a review of all the objectives of the program and the results of the shear spinning effort and biaxial testing, the 18NiCoMo (300) maraging steel was selected for Phase II and III. Material procurement for Phase II was immediately initiated to minimize the effects of a normally long lead time for this material. Concurrently, a second statistical sample was evaluated to establish the processing technique to be used in the fabrication of Phase II subscale rocket motor case.

PROCUREMENT, DESIGN, AND TOOLING FOR PHASE II

I

Concurrently with much of the latter effort in Phase I there was considerable work accomplished regarding the procurement of material and forgings for Phase II, design of two subscale pressure vessels and the design and procurement of necessary tooling.

Negotiations with several forging vendors were held and placement of an order for forgings was made with the Ladish Company of Cudahy, Wisconsin. Raw material is to be supplied by Allegheny-Ludlum Steel Corporation, Watervliet, New York. A total of three heats of material will be used to establish heat to heat variations for the production material and as well as could be arranged these three heats will reflect a range of chemical analyses. Quality control procedures were established between Lycoming, Ladish Company, and Allegheny-Ludlum for the concurrent acceptance of material at each step of the process from raw material to final forging. A tentative material specification was agreed upon to control chemistry and mechanical property capability. Present delivery schedule for finished forgings to Lycoming is the first week of June.

Page 16.

In the fabrication studies of Phase II for a subscale integral rocket motor case it is intended that several cylindrical biaxial test specimens be fabricated to the established process. These specimens, similar to the 4" tubes of Phase I, will be used to verify the results of Phase I as applied to a 15" scale model. This biaxial test specimen is shown in Figure 17. The establishment of actual manufacturing approachs for integral motor cases will be conducted in the fabrication of the 15" diameter subscale chamber and closure depicted in Figures 18 and 19. This latter subscale vessel is presently undergoing stress analysis to affix the specific dimensional criteria to make it a valid hydrostatic test vessel when successfully made.

Necessary tooling for the manufacture of the biaxial test specimens and subscale pressure vessels have been designed and orders placed for procurement. These involve the Hydrotest rig assembly shown in Figure 20; the shear spinning mandrel depicted in Figure 21. The mandrel has been completed and delivered to Lycoming. Shear spinning rollers are due in the week of 16 April 1963 and hydrotest fixtures are complete and in house.

WORK SCHEDULED FOR NEXT HALF YEAR

During the next interim reporting period the manufacturing studies for fabricating an integral rocket motor case will be conducted. Initially, the fabrication of 15" biaxial test specimens (tubes) will be accomplished to the processing cycle established in Phase I. These will be hydrostatically tested to destruction to verify the processing cycle regarding meeting the contractural requirements of 300,000 psi burst strengths. Work will then be conducted toward fabricating a <u>subscale</u> integral motor case (without welds) for subsequent verification of ultimate strength properties. TABLE I

STATISTICAL SAMPLE I - H11

Variable*				
Sclution Temp. (^o F.)	1925	1875	1825 60	1775 30
Deform. Temp. (^o F.)	1000	006	750	é00
% Reduction	0	0	0	0
Tempering Temp. (^{O.F.})	1000	006	700	500
Solution Temp. (^o F.)	1825	1775	1925	1875
Solution Time (min.)	30	60	06	120
Deform. Temp. (^o F.)	1000	006	750	600
% Reduction	25	25	25	25
Tempering Temp. (^o F.)	006	1000	500	200
Solution Temp. (^o F.)	1775	1825	1875	1925
Solution Time (min.)	60	120	30	60
Deform. Temp. (^{o.F.})	1000	006	750	600
% Reduction	50	50	50	50
Tempering Temp. (^o F.)	200	500	1000	006
Solution Temp. ^(o.F.)	1875	1925	1775	1825
Solution Time (min.)	60	30	120	06
Deform. Temp. (^o F.)	1000	006	750	600
% Reduction	75	75	75	75
Tempering Temp. (^{o.F.})	500	200	900	1000

*Constant:

Quench Rate - Air Cool Subzero Cool - 100⁰F./3 Hr. Tempering Time - 2 Hrs. plus 2 Hrs. Number Passes - 1

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STATISTICAL SAMPLE I - AM 355

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	10 1600 137 90 60 3 00 150 R 0 0 0 50 750 65	75 1900 171 60 90 12 00 150 R 25 25 2 50 650 75	00 1710 190 20 30 6 00 150 R 50 50 83	00 1375 160 30 120 5 00 150 R 75 75 50 850 99
	1900 171	137	1375 160	1710 190
	120 9	30 137	90 12	60 3
	450 30	450 30	450 30	450 30
	0 85	25 2	50 5	75 7
	950 85	850 95	750 65	650 75
Variable*	Solution Temp. ^{(o} F.)	Solution Temp. ^{(o} F.)	Solution Temp. (^O F.)	Solution Temp. (^o F.)
	Solution Time (min.)	Solution Time (min.)	Solution Time (min.)	Solution Time (min.)
	Deform. Temp. ^{(o} F.)	Deform. Temp. ^{(o} F.)	Deform. Temp. (^O F.)	Deform. Temp. (^o F.)
	% Reduction	% Reduction	% Reduction	% Reduction
	Tempering Temp. ^{(o} F.)	Tempering Temp. ^{(o} F.)	Tempering Temp. (^O F.)	Tempering Temp. (^o F.)

Quench Rate - Air Cool Subzero Cool - 100⁰F./3 Hr. Tempering Time - 3 Hrs. Number Passes - 1 *Constant:

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TABLE III

STATISTICAL SAMPLE I - 18NiCoMo

	1400 30 RT 0 700	1600 120 RT 25 800	1700 60 8T 50 900	1500 90 RT 75 1000
	1500 60 200 800	1700 90 200 25 700	1600 30 200 50 1000	1400 120 200 75 900
	1600 90 400 900	1400 60 400 25 1000	1500 120 400 50 700	1700 30 400 800
	1700 120 500 1000	1500 30 500 25 900	1400 90 500 800	1600 60 500 75 700
Variable*	Solution Temp. (^o F.) Solution Time (min.) Deform. Temp. (^o F.) % Reduction Tempering Temp. (^o F.)	Solution Temp. (^o F.) Solution Time (min.) Deform. Temp. % Reduction Tempering Temp. (^o F.)	Solution Temp. (^o F.) Solution Time (min.) Deform. Temp. (^o F.) % Reduction Tempering Temp. (^o F.)	Solution Temp. (^o F.) Solution Time (min.) Deform. Temp. (^o F.) % Reduction Tempering Temp. (^o F.)

Quench Rate - Air Cool Subzero Cool - 100⁰F./3 Hr. Tempering Time - 3 Hrs. Number Passes - 1

*Constant:

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CHEMICAL COMPOSITION

Fe		Bal.	
N2		. 084	
v	. 45		
°C	1		9.02
\mathbf{Sn}			.014
Zr			. 024
Ni	.10	4.20	18.80
Pb			.001
Ti			. 61
B			. 0026
Al		1	. 061
Мо	1. 29	2.56	5.03
Сг	4.95	15.0	. 015
Si	. 85	.35	. 10
S	. 006	.011	. 006
Ъ	. 005	.011	. 004
Мп	. 35	1.01	.014
υ	.38	. 13	. 026
Material	Н-11	AM 355	18NiCoMo

MECHANICAL PROPERTIES*

	Ultimate Tensile	Yield Strength		Reduction	
Material	Strength (ksi)	at 0.2% offset (ksi)	Elengation (% 4D)	of Area (%)	Hardness Rc
H-11	288. 6	251.2	8 8 8	17.9	54
AM 355	178.4	162.8	17.0	48.1	47
18NiCoMo	286. 2	278.2	7.0	30.7	52

*Results of (3) tests.

Heat Treatments: H-11 -

- Austenitized 1825⁰F., 1 hr., air cooled, subzero cooled to -100⁰F., 3 hrs., double tempered 1025⁰F. for 2 hrs. each. I
- Solution treated 1710^{0} F., 1 hr. air cooled, subzero cooled to -100^{0} F., 3 hrs., temper 1000^{0} F., air cooled. ł AM 355
- Solution treated 1500°F., 1 hr., maraged 900°F., 3 hrs., air cooled. 18NiCoMo -

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	Hardness Rc	23	2 23	57	60	3	99	57	:	62	!	99	:	 99	60	 -	ø	:		:			:	:	ļ	:	
	Total Gross Strain (%)			Shattered	Shattered	Shattered	Shattered	Shattered		0.1		1.0		Shattered	Shattered	 											
	Nominal Yield Strength (0. 2% offset) (ksi)	290	307	279	*	*	297	327		392	!	*	:	316	365	 ;		:		:	;		:	:	:	:	
	Nominal Ultimate Strength (kai)	356	333 362	338	380	233	322	394	1	413	!	319	4 1 1 4	378	388	!		!		ł	!		ł	!	!	;	
	Double Temper at (^O F.)	1000	00 00- 200	500	006	1000	200	100	Spin	200	Spin	200	1000	1000	906	ted	ice cracks	bei	tce cracks	Spin	ted	ice cracks	Spin	Spin	Spin	Spin	Spin
	Subzero Cool at - 100°F. (Hre.)	3	m m	9	3	e	ñ	e	Did Not	ر	Did Not	6	•	ŝ	e	Spun but exhibit	numerous surfa	Spun but exhibit	numerous surfa	Did Not	Spun but exhibit	numerous surfa	Did Not				
3urst Tube Test Results	Shear Spin Blank Temperature at Start of Spinning (^o F.)	:		:	1000	006	750	290	1000	1000	906	910	740	750	600	1000		1000		920	006		750	760	600	600	909
arameters and I	Mandrel Temperature (°F.)	:	; ;		1000	006	750	009	1000	1000	006	910	740	750	600	1000		1000		920	906		750	760	620	009	600
l Process P	Solution Time (min.)	120	06 9	30	30	60	6	120	 90	90	120	120	30	30	99	 60		60		8	30		120	120	6	96	6
н-1	Solution Temperature (^O F.)	1925	1875	1775	1825	1775	1925	1875	 1775	1775	1825	1825	1875	1875	1925	 1875		1875		1925	1925		1775	1775	1825	1825	1825
	Number of Passes	0		• •	1	-	-	1	 1	-	-	-	I	-	-	 2		N	_	2	2		1	~	2	~	2
	Wall Reduction (%)	· 0	0 0		25	52	25	25	 2	8	8	50	2	20	3	75		75		75	75		75	75	75	75	75
	Wall Reduction (in.)	0	0 0		0,060	0,060	0,060	0.060	0.375	0. 188	0. 188	0. 188	0. 188	0. 188	0. 188	0.414		0.414		0.414	0.414		0.414	0.414	0. 555	0.414	0.414
	Sample Number (S/N)	6	<u>8</u>	21		,	16	12	2	65		3	67	13	1	78		1 0		~	17		و.	1 76	15	*	

*Not Determinable

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TABLE VI

AM 355 Process Parameters and Burst Tube Test Results

	r	T		T
Hardness Rc	4-94 19-64 19-14	48-49 45-47 45-51	49-53	
Total Gross Strain (%)	10.5 2.2 7.1	7.8 10.0 2.1	9, 5	
Nominal Yield Strength (0.2% offset) (ksi)	199 222 208 201 201	248 230 225	542 1911 - 192	
Nominal Ultimate Strength (ksi)	274 247 265 249	296 252 279	330 5 68	
Tempered at (°F.)	950 850 650	850 950 650 Spin	Spin Spin Spin 650 850 850	niq Quanta niq Niq Niq Niq Niq Niq Niq Niq Niq Niq N
Subzero Cool at - 1000F. (Hrs.)	~~~~	3 3 Did Not	Did Not Did Not Did Not Did Not Did Not	Did Not Did Not Did Not Did Not Did Not Did Not
Shear Spin Blank Temperature at Start of Spinning (^o F.)	::::	450 300 160 R.T	450 450 300 150 8.T	450 300 150 8.1
Mandrel Temperature (°F.)		450 300 160 RT	450 450 300 150 8.T	450 450 300 150 RT
Solution Time (min.)	120 90 30	30 60 120	90 90 30 30 60	66 86 12 12 12 12 12 12 12 12 12 12 12 12 12
Solution Temperature (°F.)	1900 1710 1600 1375	1600 1375 1710 1710	1375 1375 1600 1710 1710	1710 1710 1900 1375
Number of Passes				NNNNN
Wall Reduction (%)		5 52 52 52 52	8 8 8 8 8 8	55555
Wall Reduction (in.)		0.060 0.060 0.060 0.060	0. 188 0. 188 0. 188 0. 188 0. 188 0. 188 0. 188	0.415 0.415 0.415 0.415 0.415 0.415 0.415
Sample Number	35 22 34 30	25 29 33	9 2 7 8 9 8	222223

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TABLE VII

18NiCoMo Process Parameters and Burst Tube Test Results

Total Gross Strain Hardness (%) Rc	5.0 51-48 3.7 54-51 6.8 45-45 5.0 49-47	1.2 53 7.0 49-51 1.0 45-45 4.8 52-53	1.1 53-55 2.8 49-49 3.8 53-53 3.2 53-53	
Nominal Yield Strength (0. 29 offset) (ksi)	310 316 283 245	348 295 213 312	343 361 1	262
Nominal Ultimate Strength (ksi)	349 333 300 272	351 31 4 236 322	348 321 321 368	\$
Maraged 3 Hrs. at (°F.)	1000 900 800	900 1000 700 800	800 700 1000 900	700 tt Spin tt Spin ell-mouth tt Spin tt Spin
Subzero Cool at -100°F. (Hrs.)	**			3 Did Nc Did Nc Excessive b part no good Did No Did No
Shear Spin Blank Temperature at Start of Spinning (oF.)		500 400 200 RT	500 400 220 RT	500 400 400 200 200 200
Mandrel Temperature (^o F.)		500 400 200 RT	500 400 220 RT	200 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
Solution Time (min.)	30 86 30	20 60 120	60 30 60 80 80 80 80 80 80 80 80 80 80 80 80 80	30 30 120 120
Solution Temperature (^O F.)	1700 1600 1500	1500 1400 1700 1600	1400 1500 1700	1400 1400 1400
Number of Passes				~~~~
Wall Reduction (%)		52 52 53 53	8888	* * * * * * *
Wall Reduction (in.)	0000	0,00,00 0,00,00 0,00,00 0,00,00	0. 188 0. 188 0. 188 0. 188	0.415 0.415 0.415 0.415 0.415 0.415
Sample Number	24 2 38 29 4 5	6 1 2 6	×314	2828 42

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TABLE VIII

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STATISTICAL SAMPLE II - 18NiCoMo (300)

	500 65 1 3	500 65 3 3	500 65 1 6	500 65 6
	500 40 1 3	500 40 2 3	500 40 1 6	500 40 6
	RT 65 1 3	RT 65 2 3	RT 65 1 6	RT 65 6
	RT 40 1 3	RT 40 2 3	RT 40 1 6	RT 40 6
Variable*	Deform. Temp. (^O F.) % Reduction Number of Passes Aging Time - Hours	Deform. Temp. (^O F.) % Reduction Number of Passes Aging Time - Hours	Deform. Temp. (^o F.) % Reduction Number of Passes Aging Time - Hours	Deform. Temp. (^o F.) % Reduction Number of Passes Aging Time - Hours

*Constant: Solution Temperature 1500⁰F. Solution Time 1 Hour Quench Rate Air Cool Subzero Cool -100⁰F. Aging Temperature 900⁰F. ł

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	Hardness Rc	53-53	55-53	53-53	52-54	53-53	56-54	55-55	55-53	55-55	54-53	15-15	55-55	55-53	54-53	55-55	53-55
	Total Gross Strain	3. 0	3.3	3. 1	3.1	2.6	4 1	1.9	2.8	2.3	4.4	3.6		4.6	1.9	3.3	2.6
	Yield Strength (0. 2% offset) (ksi)	337	348	337	333	332	367	327	369		350	356	:		:	344	;
le)	Nominal Ultimate Strength (ksi)	341	351	353	342	337	369	334	377	344	351	359	183	383	364	346	349
ond Statistical Samp	Maraging Time at 900°F. (Hrs.)	8	3	¢	ę	3	3	¢	9	E	ŋ	9	9	e	3	9	·J
Tube Test Results (Seco	Shear Spin Blank Temperature at Start of Spinning (oF.)	RT	RT	RT	RT	500	500	500	500	RT	RT	F.R.	RT	200	500	500	200
neters and Burst	Mandrel Temperature (⁰ F.)	RT	RT	RT	RT	500	500	500	500	RT	RT	RT	RT	500	500	500	200
cess Paran	Solution Time (min.)	99	60	60	60	60	60	60	60	99	60	99	. 99	99	99	09	60
I8NiCoMo Pro	Solution Temperature (°F.)	1500	1500	1500	1500	1500	1500	1500	1500	1500	1500	1500	1500	1500	1500	1500	1500
	Number of Passes	-	8	-	8	-	~	-	2	-	2	-	2	-	2		N
· · · ·	Wall Reduction (%)	9	40	\$	9	4	\$	40	\$	\$3	65	6 5	5 9	65	ę2	65	65
	Wall Reduction (in.)	0. 160	0. 160	0. 160	0. 160	0. 256	0.256	0. 256	0. 256	0.260	0. 376	0. 256	0. 376	0. 256	0. 256	0, 256	0.376
	Sample Number	5	3	18	3	08	462	8 3	82	84	22	85	71+	e	5	8	\$69

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TABLE IX

*Contained internal defects.

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Figure 2. - Phase I Laboratory Burst Tube

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Assembled Subscale Fressure Vessel Installed in Hydrostatic Test Stand Figure 3.



Figure 4. Biaxial Stress-Strain Curves for H-11 Steel Pressure Vessels

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Neg. No. 12452-E

Mag: 3/4X



Neg. No. B8000-6

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Mag: 10X

Figure 5. Biaxial Test Specimen Nr. 66 of H-11 Steel Showing Typical Ductile Failure



Neg. No. 8856-B

Mag: 3/4X



Neg. No. 8858-B

Mag: 10X

Figure 6. Biaxial Test Specimen Nr. 10 of H-11 Steel Showing Typical Ductile Failure



Neg. No. 8856-C

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Mag: 5/8X



Neg. No. 8858-C

Mag: 10X

Figure 7. Biaxial Test Specimen Nr. 13 of H-11 Tool Steel Showing Typical Brittle Failure



Neg. No. 8848

Mag: 10X

Figure 8. Biaxial Test Specimen Nr. 14 of H-11 Tool Steel Showing Typical Brittle Fracture



Figure 9. Biaxial Stress-Strain Curves for AM 355 Stainless Steel Pressure Vessels



Neg. No. 8854

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Mag: 3/4X

Figure 10. Biaxial Test Specimen Nr. 22 of AM 355 Stainless Steel in Test Fixture Immediately After Failure



Neg. No. 8846-A

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Mag: 3/4X



Neg. No. 8851-AMag: 102Figure 11. Biaxial Test Specimen Nr. 25 of AM 355 Stainless
Steel Showing Typical Ductile Failure



Neg. No. 8852

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Mag: 3/4X



Neg. No. 8858-G

Mag: 10Y

Figure 12. Biaxial Test Specimen Nr. 24 of AM 355 Stainless Steel Showing Typical Ductile Failure



Vessels

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Neg. No. 8846-B

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Mag: 3/4X



Neg. No. 8851-B

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Mag: 10X

Figure 14. Biaxial Test Specimen Nr. 36 of 18NiCoMo Steel Showing Typical Ductile Failure





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Neg. No. 8019-12

Mag: 3/4%

Neg. No. 8019-35

M.ag: 102

Figure 16. Biaxial Test Specimen Nr. 88 Which Exhibited Highest Biaxial Strength (383,000 psi) in 18NiColvio Steel.

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Phase II Biaxial Test Specimen

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Figure 20. .

Hydrotest Rig Assemb

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DISTRIBUTION LIST

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