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The Effects of Cold Rolling on the Mechanical Properties of Type 310 Stainless Steel at Room and Cryogenic Temperatures

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The Effects of Cold Rolling on the Mechanical Properties of Type 310 Stainless Steel at Room and Cryogenic Temperatures

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J. L. Christian, J. D. Gruner and L. D. Girton

#### ABSTRACT

N66-22934

The purpose of this investigation was to determine the applicability of cold rolled Type 310 stainless steel for structural uses at cryogenic temperatures. Yield and tensile strengths, elongation and notched toughness were determined as a function of cold rolling from 0 to 92% reduction and of temperatures from 78° to -423°F.

The results indicate that high strengths may be achieved by cold rolling and that the toughness is adequate for structural applications at -423°F for the 0 - 85% cold rolled tempers. An evaluation was also made on the room temperature formability of annealed and cold rolled 310 stainless steel and was found to be acceptable for the 0 - 37.5% cold rolled tempers. As a result of this, and previous investigations, it is believed that cold rolled Type 310 stainless steel is an excellent material for structural applications at cryogenic temperatures.

Author

#### INTRODUCTION

The advent of the Space Age has placed an increasing demand upon materials' engineers to provide materials with improved properties for construction of missiles and space vehicles. The properties of foremost importance are high strength, in particular the strength-to-density ratio, and adequate toughness over the entire spectrum of the operating environments. In addition, the materials must be weldable and formable, posses adequate resistance to corrosion, and be readily available in various forms and sizes at a reasonable cost. The necessity for these properties is due to the immediate need of reliable, lightweight, inexpensive flight vehicles to achieve the goals of the national space program.

Toughness, as used herein, is defined as the ability of a material to resist brittle failure under severe conditions of loading, stress concentrations and temperature. Normal loading conditions may include bi- and tri- axial loading, vibrational or impact loading, as well as very high operating stresses (up to 90% of the yield strength of the material). Stress concentration may be caused by several factors. These include non-axial loading, discontinuities (such as found at welds), mechanical joints and re-entrant angles of formed parts, surface imperfections caused by machining, tooling marks and handling, and discontinuities such as inclusions, stringers and large intermetallic precipitates within the material. Depending upon the vehicle, and location within the vehicle, temperatures may vary from very high temperatures (e.g., leading edges of reentry vehicles, engine components, etc.) to very low temperatures. The very low temperatures result from the use of liquid oxygen (-297°F) and liquid hydrogen (-423°F) as propellants and under certain conditions in outer space. Due to the extensive use of liquified gases as propellants and because materials are inherently more brittle at lower temperatures, the knowledge of the properties of structural materials at cryogenic temperatures is of prime importance.

One class of materials which appears to possess the requisite properties for missile and space vehicle structures is the cold rolled austenitic stainless steels. As a result, extensive investigations have been conducted to determine the properties of the 300 series stainless steels (Ref. 1-10). In particular, and primarily as a consequence of its use in the Atlas and Centaur vehicles, Type 301 stainless steel has received considerable attention and has been evaluated as a function of form, temper (amount of cold work), and temperature. The results of these investigations have shown that cold rolled (1/4 hard to extra full hard) Type 301 stainless steel possesses excellent properties for liquid oxygen (-297°F) tankage and therefore is ideal for construction of vehicles, such as Atlas, which are fueled with RP-1 (high grade kerosene) and liquid oxygen. However, it has been found that many heats of cold rolled Type 301 stainless lack adequate toughness for welded structures at the temperature of liquid hydrogen, -423°F (Ref. 1, 4, 10). The reasons for the partial embrittlement of 301 at -423°F is primarily attributed to the presence of martensite, in particular that martensite which transforms from the meta-stable austenite as a result of plastic deformations at cryogenic temperatures. (Ref. 10,11).

Because of the partial embrittlement of 301 at -423°F, attention has been focused on the higher nickel, and therefore more stable, stainless steels such as Types 304 and 310. Type 310 stainless steel appears to be the most promising since it has been found to be fully stable (no occurrence of martensitic transformation) due to cold rolling or application of stress (to failure) at -423°F (Ref. 4, 10, 12). The primary disadvantages of 310 as compared to 301 are a lower strength to density ratio of the extra full hard cold rolled material and a decrease in the formability of the 1/4 to 3/4 hard cold rolled material. The purpose of this investigation was, therefore, to determine the properties, primarily strength, toughness and formability, of Type 310 stainless steel as a function of cold rolling and temperature.

#### MATERIALS AND PROCEDURE

Two heats of Type 310 stainless steel were evaluated in this investigation. The chemical analysis of these heats is given in Table I. The cold rolling of heat 84074 and of the 75% CR condition of heat 43631 was performed by the suppliers. The remainder of the cold rolling was performed at room temperature on a six inch wide, two high - four high laboratory rolling mill at GD/A.

Drawings of the tensile and notched tensile specimens are shown in Figure 1. The notched tensile specimens had a stress concentration factor  $(K_t)$  of 6.3 as determined by  $\sqrt{a/r}$ , where a is equal to 1/2 of the width between the notches and r is the radius at the root of the notch. Tensile tests were performed at 78°F (room temperature), -100°F by immersion in a bath of dry ice and alcohol, -320°F by immersion in liquid nitrogen and -423°F by immersion in liquid hydrogen. A full description of the testing apparatus and experimental procedure is given in Reference 13.

As an indication of the approximate formability of the cold rolled 310 stainless steel, standard ductility cup tests were performed at room temperature. The test consisted of drawing a cup in the metal by a 1.5 inch diameter hemispherical punch which is hydraulically activated. The hydraulic pressure and cup height are determined by means of pressure and dial gauges upon onset of failure. Specimen sizes and typical failures are shown in Figure 10.

#### DISCUSSION OF RESULTS

The results of the tensile and notched tensile tests are given in Table 2 and Figures 2-4. Although only the longitudinal data are presented in the figures, there does not appear to be much of a directional effect in Type 310 stainless steel as a function of cold rolling or temperature. As would be expected, the yield and tensile strengths increase with an increase in the amount of cold rolling and with a decrease in the testing temperature. Type 310 stainless steel has been found to be a fully stable austenitic material with no martensitic transformations occurring as a result of cold working or thermal cycling (Ref. 10, 11). Therefore, the increase in strength which results from cold rolling is due solely to the work hardening of the austenite and thus explains why much higher strengths ( $F_{ty}$  of 180-200 Ksi and  $F_{tu}$  of 200-240 Ksi at 78°F) are obtained by cold rolling Type 301 stainless steel which undergoes an austenite to martensite transformation (Ref. 1, 4, 10). The large increase in tensile strength with reduction in temperature (60-100% increase from 78° to -423°F) are typical of face-centered cubic lattice (austenitic) structures. However, the large increases in yield strength, 60-180% from 78° to -423°F, are atypical (Ref. 14, 15). Similar behavior has previously been noted for some high strength aluminum alloys and is explained on the basis of a highly strained lattice structure which results from the large amount of alloying elements present in solid solution (Ref. 16).

The elongation of Type 310 stainless steel decreases with an increase in the amount of cold rolling, but increases with a reduction in temperature, at least to  $-320^{\circ}F$  with a small decrease from  $-320^{\circ}$  to  $-423^{\circ}F$ . At room temperature the elongation decreases rapidly from the annealed (40% elongation) to the 40% cold rolled condition (about 5% elongation), and then gradually decreases to about 1-2% elongation for the 90-92% cold worked material. At  $-320^{\circ}F$  the elongation is much greater for the annealed material, about 70%, and decreases in a more linear manner, to about 10%, for the 80% cold rolled condition. At  $-423^{\circ}F$  the elongation decreases nearly linear from 50% for the annealed material to about 5% for the 92% cold rolled material. Based upon elongation, the ductility of annealed or cold rolled Type 310 stainless steel is greater at cryogenic temperatures than at room temperature.

It has been found that the notched tensile strengths and resulting notched/ unnotched tensile strength ratios provide a good indication of a material's toughness or resistance to brittle failure (Ref. 10, 12). Analysis of the notched tensile data indicate the following. At room temperature, the notch toughness is slightly improved by cold rolling to about 30-40% reduction and then gradually decreases upon further cold rolling so that beyond 90% reduction the notched/unnotched tensile strength ratio is below unity. At -320°F, the same improvement in notch toughness by cold rolling to about 40% reduction is witnessed, and the toughness does not appear to be further affected upon cold rolling to 80% reduction. At -423°F, the notch toughness is improved by cold rolling to about 75% reduction; however, a sharp decrease in the notched tensile strength beyond 80% reduction results in a significant decrease in the notched/unnotched tensile strength ratio with values below unity resulting beyond about 87% reduction. A possible explanation for this decrease in toughness at -423°F is due to the presence of a large number of carbide stringers in the severely cold worked material (see Figures 6-9). It is recommended that 310 stainless steel which is to be used for structural applications at -423°F not be cold rolled beyond about 80% reduction.

Mechanical properties of fusion and resistance welds of cold rolled 310 stainless were not determined in this study due to the lack of material; however these properties have been thoroughly evaluated for the 75% cold rolled condition and are reported in References 4 and 10.

Another property of the material which was required in order to evaluate its applicability for liquid hydrogen and liquid oxygen tankage was formability, in particular the ability of the alloy to be stretch formed into gore or pie sections which are subsequently welded to make bulkheads (see Figure 11). Because of material limitations and in order to minimize costs, cup tests were used to evaluate the formability of annealed and cold rolled Type 310 stainless steel. The data obtained on five conditions of 310 are given in Table 3 and Figure 5. In addition, cupping test data are also presented for four cold rolled conditions of Type 301 stainless steel. From the vast amount of experience obtained in stretch forming Type 301 stainless steel for the Atlas and Centaur bulkheads, it has been found that 1/2 hard and 3/4 hard 301 may be easily stretch formed over the most severe bulkhead forming tool which has a 60-inch by 30-inch compound radius. On the other hand, the extra full hard 301 cannot be successfully stretch formed, and the full hard 301 is stretch formable only to a limited extent involving mild curvatures. From this information and the data in Table 3, it was ascertained that a cup height of 0.350 inches or greater at failure, using a 1.5 inch diameter hemispherical punch, was required in order to have the desired formability.

From the cupping test data it appears that 0-37.5% cold rolled 310 stainless possesses adequate formability to meet the stretch forming requirements. Because of the limited width of the material rolled on the laboratory mill, it was not possible to actually stretch form some of the 37.5% cold rolled material. However, several sheets of 60% cold rolled 310 were partially annealed by heating to 1400-1500°F, and the resultant tensile properties approximated those for the 10 to 20% cold rolled material. These sheets were successfully stretch formed and are shown in Figure 11. Although the cupping tests were performed primarily to determine the ability of the material to be stretch formed, it is believed that these data may also be useful for evaluating other forming applications.

#### SUMMARY

The tensile and notched tensile properties were determined on Type 310 stainless steel as a function of temper (0-92% reduction by cold rolling) and temperature (78°, -320° and -423°F). Also, the formability of several tempers of Type 310 stainless steel at room temperature was evaluated by means of cup tests. From the data obtained in this investigation the following conclusions and recommendations are made:

- The yield and tensile strength of 310 stainless steel increases with an increase in the amount of cold work and a decrease in the testing temperature.
- 2. The ductility, as measured by elongation, decreases with increased amount of cold working. However, the ductility at cryogenic temperatures is greater than at room temperature for 310 stainless steel at a given temper.
- 3. The toughness, as determined by notched tensile strengths and resulting notched/unnotched tensile strength ratio, is not severely affected by cold work at 78° or -320°F but is significantly impaired at -423°F for tempers beyond 80-85% cold work.
- 4. The formability, as determined by cup tests, is decreased by increased amounts of cold rolling, but remains comparable to 1/2 and 3/4 hard Type 301 stainless steel to about 35-40% reduction by cold rolling.
- It is recommended that Type 310 stainless steel cold rolled beyond 85% reduction not be used for structural applications at -423°F.

#### ACKNOWLEDGEMENTS

The authors wish to acknowledge the assistance of their associates who contributed to this paper and, in particular, to A. Hurlich, who supplied technical counsel throughout the course of this investigation. The work was performed under the sponsorship of General Dynamics/Astronautics, whose permission to publish this paper is gratefully acknowledged.

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## TABLE 1

## Chemical Analysis of Type 310 Stainless Steel

	310 CRES 40-75% C.R.	310 CRES 0-92% C.R.	
Heat No.	84074	43631	
Coil No.	-	44942	
Supplier	Allegheny-Ludlum	Washington	
Gauge (In.)	0.020	0.006-0.080	
Content (Wt. %)			Specification AMS 5521B
C	0.04	0.060	0.08 Max.
Cr	25.26	24.62	24.0-26.0
Cu	-	0.23	0.50 Max.
Fe	Bal.	Bal.	Bal.
Mn	1.66	1.60	2.00 Max.
Мо	-	0.32	0.50 Max.
Ni	19.58	19.66	19.0-22.0
Ρ	-	0.030	0.040 Max.
S	-	0.011	0.030 Max.
Si	0.70	0.58	0.75 Max.

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TABLE 2

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### Mechanical Properties of Type 310 Stainless Steel at Various Degrees of Cold Rolling

Heat No.	% Cold Work	Test Temp.(°F)	Grain Direct.	Fty (KSI)	F <sub>tu</sub> (KSI)	Elong.	Notch T.S. K <sub>t</sub> = 6.3 (KSI)	Notched/Unnotched Tensile Ratio
43631	0	78	Long.	40.3	85.8	46	93.9	1.09
		78	Trans.	34.8	86.8	46	91.6	1.05
		-320	Long.	86.5	164	71	155	0.95
		-320	Trans.	89.8	165	71	151	0.92
		-423	Long.	113	196	5 <b>2</b>	182	0.93
		-423	Trans.	117	197	44	179	0.91
43631	12.5	78	Long.	87.6	100	24	115	1.15
		78	Trans.	88.9	101	22	120	1.19
		-320	Long.	131	182	51	186	1.02
		-320	Trans.	123	180	54	187	1.04
		-423	Long.	156	221	43	213	0.96
		-423	Trans.	144	215	42	211	0.98
4363 <b>1</b>	37.5	78	Long.	<b>12</b> 5	139	9	167	1.20
		78	Trans.	126	140	9	167	1.19
		-320	Long.	162	208	31	237	1.14
		-320	Trans.	165	208	31	235	1.13
		-423	Long.	191	243	27	261	1.07
		-423	Trans.	187	243	32	263	1.08
84074	40	78	Long.	138	153	4	168	1.10
		-100	Long.	156	170	13	-	-
		-100	Trans.	151	180	12	-	-
		-320	Long.	182	221	24	236	1.07
		-423	Long.	200	276	28	276	1.00
			Trans.	<b>2</b> 18	263	25	294	1.11
84074	60	78	Long.	153	174	3	160	1.03
		-320	Long.	205	233	17	248	1.06
		-423	Long.	232	278	18	296	1.06
			Trans.	226	285	16	308	1.08
43631	62.5	78	Long.	161	167	4	181	1.09
	-	78	Trans.	150	181	5	181	1.00
		- 320	Long.	210	236	18	263	1.11
		-320	Trans.	197	247	16	297	1.20
		-423	Long.	240	270	10	296	1.10
		-423	Trans.	224	280	16	321	1.15
84074	75	78	Long.	160	179	3	197	1.10
		- 320	Long.	215	242	13	269	1.11
		-423	Long.	<b>2</b> 54	281	14	312	1.11
		-423	Trans.	247	298	2	301	1.01

### TABLE 2 (Continued)

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## Mechanical Properties of Type 310 Stainless Steel at Various Degrees of Cold Rolling

Heat No.	% Cold Work	Test Temp.(°F)	Grain Direct.	Fty (KSI)	$\frac{F_{tu}}{(KSI)}$	Elong. 	Notch T.S. Kt = 6.3 (KSI)	Notched/Unnotched Tensile Ratio
43631	75	78 78 -100 -100 -320 -320 -423	Long. Trans. Long. Trans. Long. Trans. Long.	157 163 190 192 223 225 261	181 199 204 225 251 273 290	2 4 3 10 11 5	194 193 220 236 278 292 329	1.07 0.97 1.08 1.05 1.11 1.07 1.12
43631	80	-423 78 78 -320 -423 -423	Trans. Long. Trans. Long. Trans. Long. Trans.	280 168 162 236 246 254 261	317 189 210 256 286 296 316	10 2 4 9 10 9 8	328 198 210 267 318 320 355	1.03 1.05 1.00 1.05 1.11 1.08 1.13
43631	85	78 -423	Long. Long.	167 253	191 300	2 7	197 307	1.03
43631	90	78 -423	Long. Long.	170 264	198 308	1 5	198 300	1.00 0.97
43631	92	78 -423	Long. Long.	169 270	<b>20</b> 0 313	<b>2</b> 5	196 290	0.98 0.93

TABLE 3

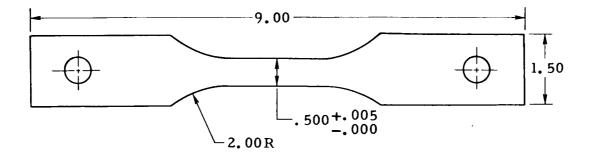
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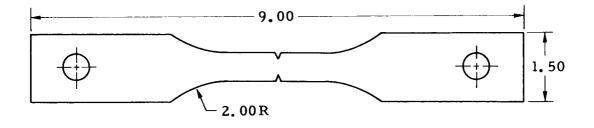
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Results of Cupping Tests on Types 301 and 310 (Heat No. 43631) Stainless Steel

5" Dia)	Minimum	0.185	0.295	0.355	0.402	0.692	0.500	0.350	0.190	0110
(in.) (1.5	Average	0.200	0.307	0.368	0.408	0.703	0.503	0.360	0.203	0.170
CUF HEIGHT (in.) (1.5" Dia)	No. of Tests	Q	Q	Q	Q	ω	Q	7	4	ħ
	El. (%)	m.≠	<b>\0</b> I	10	20 5¢	40 47	22 22	σσ		01- <del>1</del>
<b>LATIES</b>	Ftu (KSI)	220 225	205 -	190 190	170 170	85.8 86.8	100 101	139 140	167 181	189 210
TENSILE PROPERTIES	Fty (KSI)	205 190	180 -	165 155	125 120	40.3 34.8	87.6 88.9	125 126	161 150	168 162
F	Direction	Long. Trans.								
	<u>Thickness (n.</u> )	0.026	0.022	0.023	0.023	0.080	0.070	0.050	0.030	0.016
	Condition	XFH ( 60% CR)	FH ( 50% CR)	3/4 H ( 40% CR)	1/2 н ( 30% ск)	Annealed	12.5% CR	37.5% CR	62.5% CR	80% CR
	Material	301 S.S.	301 S.S.	301 S.S.	301 S.S.	310 S.S.				





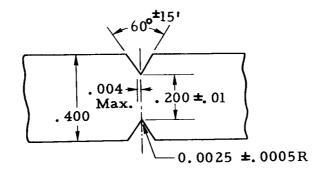
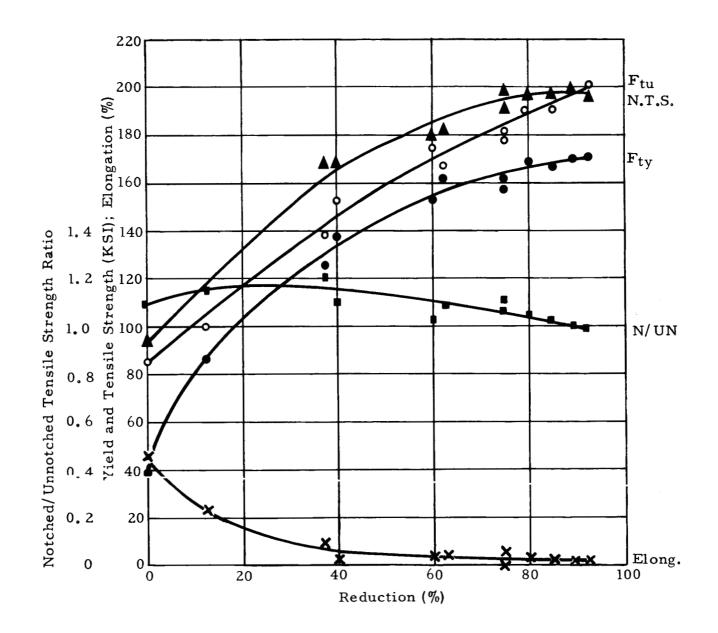


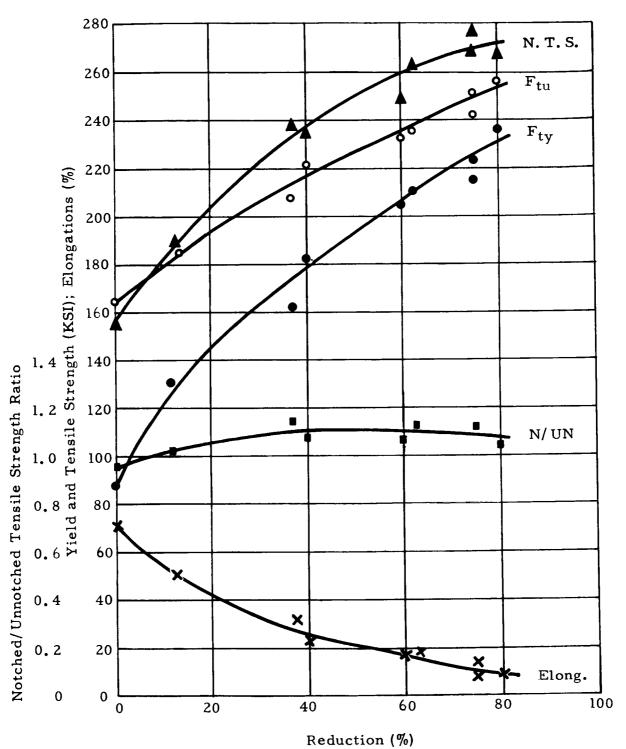
Figure 1. Tensile specimens for cryogenic testing (all dimensions in inches).

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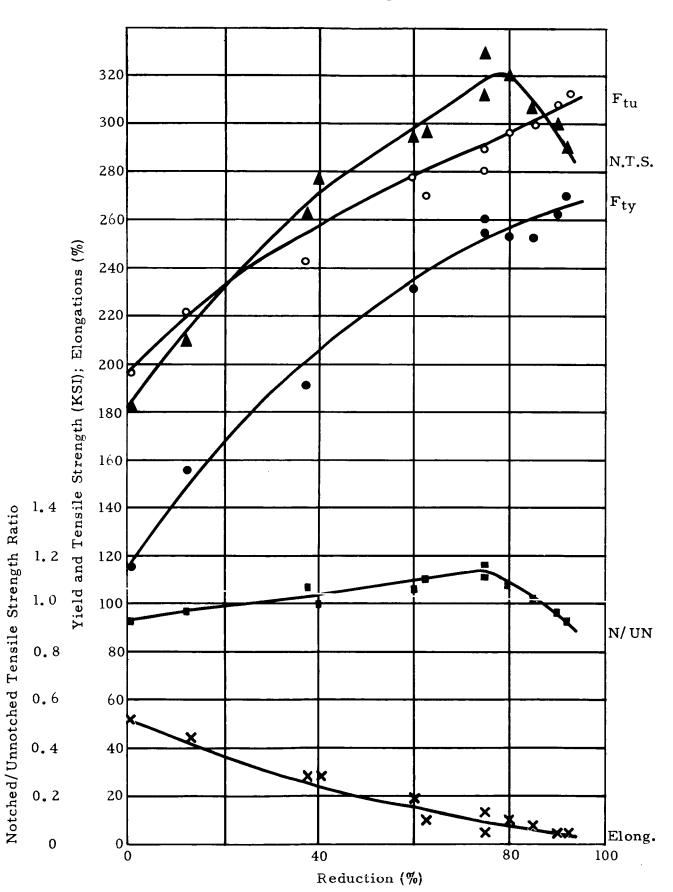


Mechanical Properties (Long. Dir.) at -320° F.

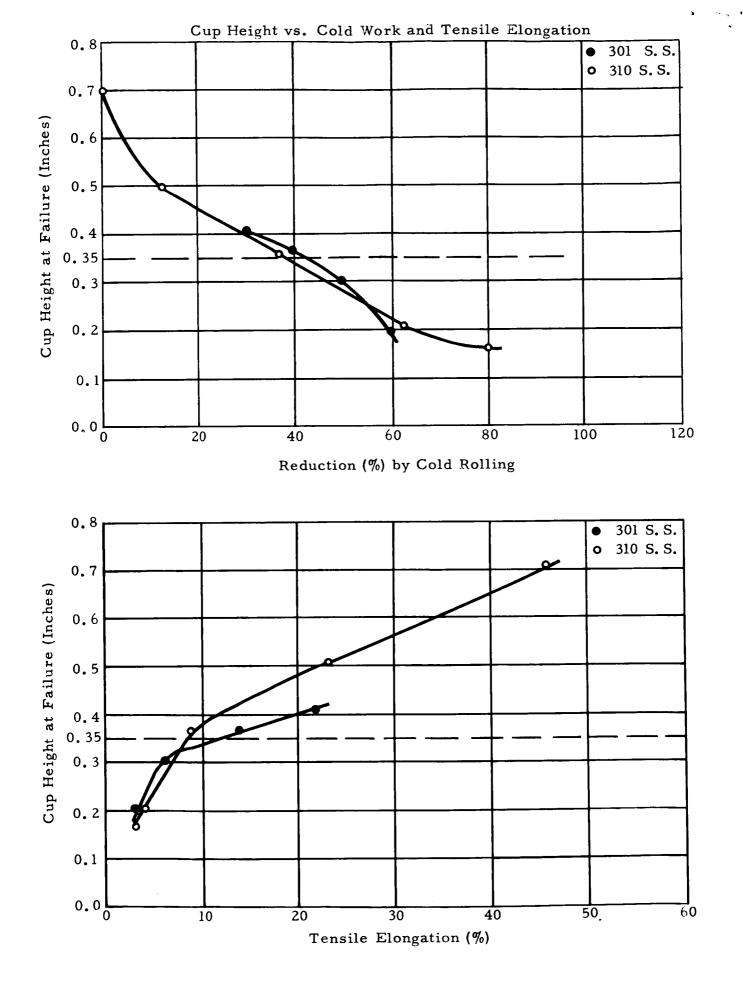
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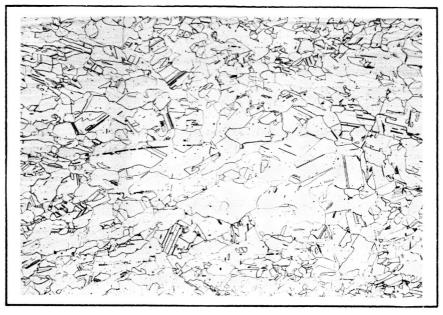
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Figure 3.



Mechanical Properties (Long. Dir.) at -423° F.

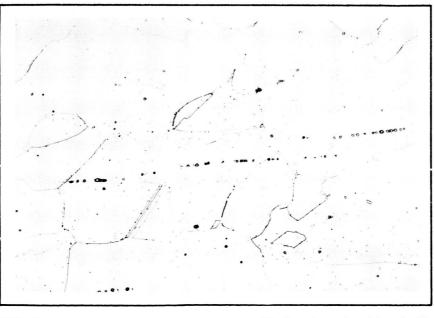






Etchant: Oxalic Acid

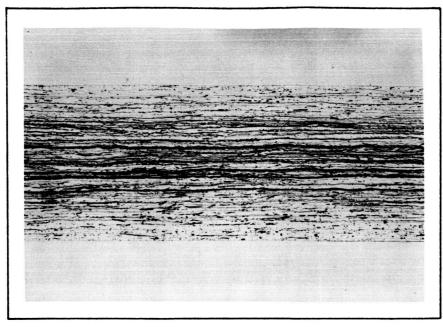
The microstructure of a longitudinal section of the annealed 0.080" thickness material. A relatively small amount of inclusions and foreign particles are visible in a recrystallized austenitic matrix.





Etchant: Oxalic Acid

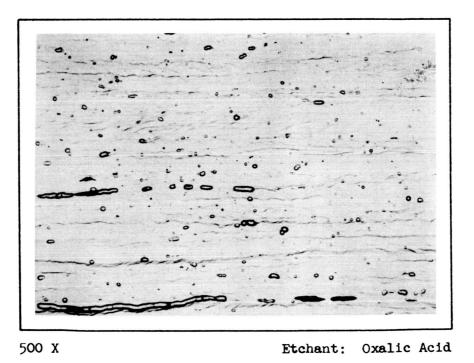
A higher magnification view of the microstructure from the annealed .090" thickness material.



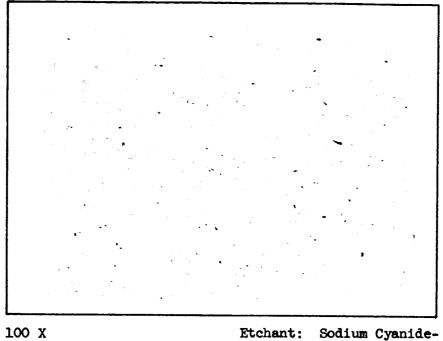


Etchant: Oxalic Acid

The microstructure of a longitudinal section of the 0.016" thickness material. This material has been severely cold worked (80% reduction)

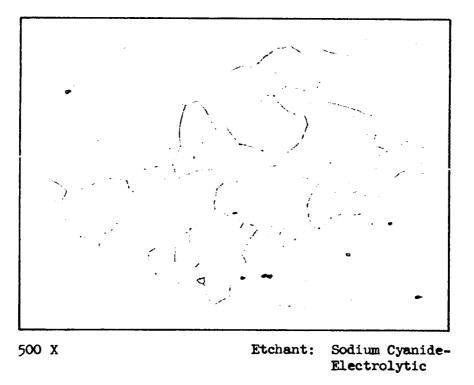


A higher magnification of the microstructure of the 0.016" thickness material.



Electrolytic

The presence of a discontinuous network of grain boundry carbides, shown as dark lines, was determined by electrolytically etching with sodium cyanide.

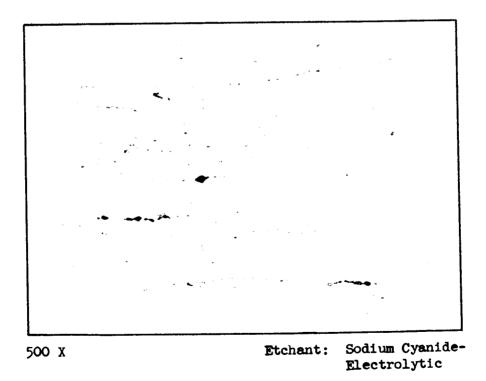


A higher magnification of the discontinuous network of grain boundry carbides revealed by the electrolytic sodium cyanide etch.

Etchant: Sodium Cyanide-100 X

A longitudinal cross section of the 0.016" material electrolytically etched with sodium cyanide. The network of globular carbides has been elongated in the direction of cold work.

Electrolytic



A higher magnification of the elongated network of globular carbides in the 0.016" material.

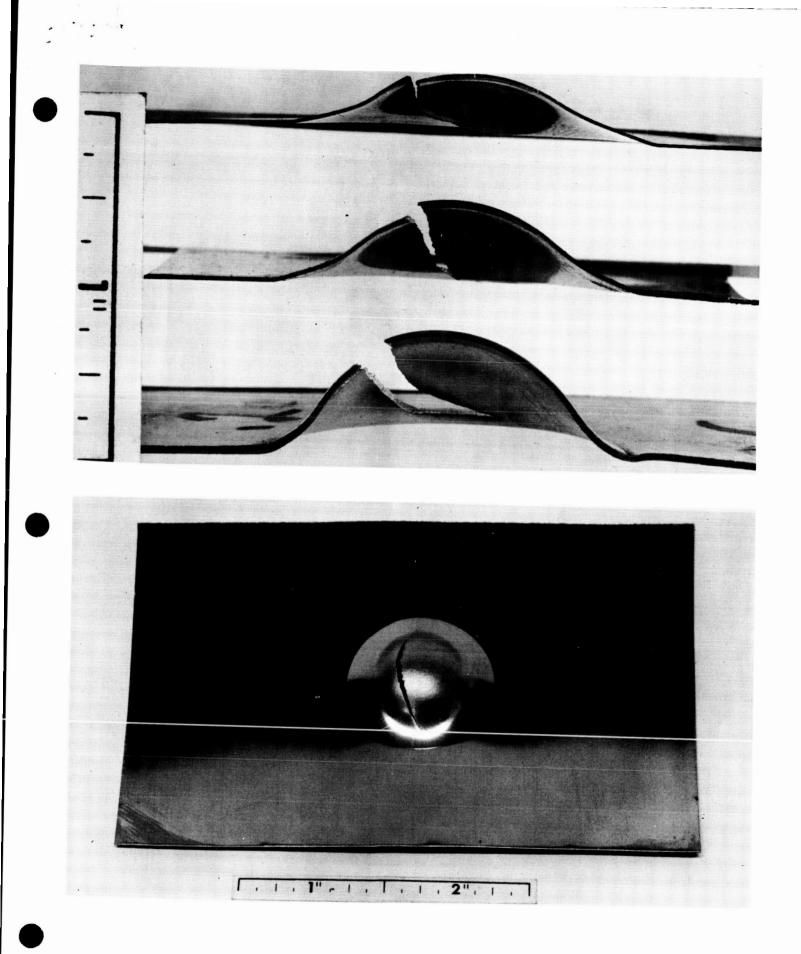
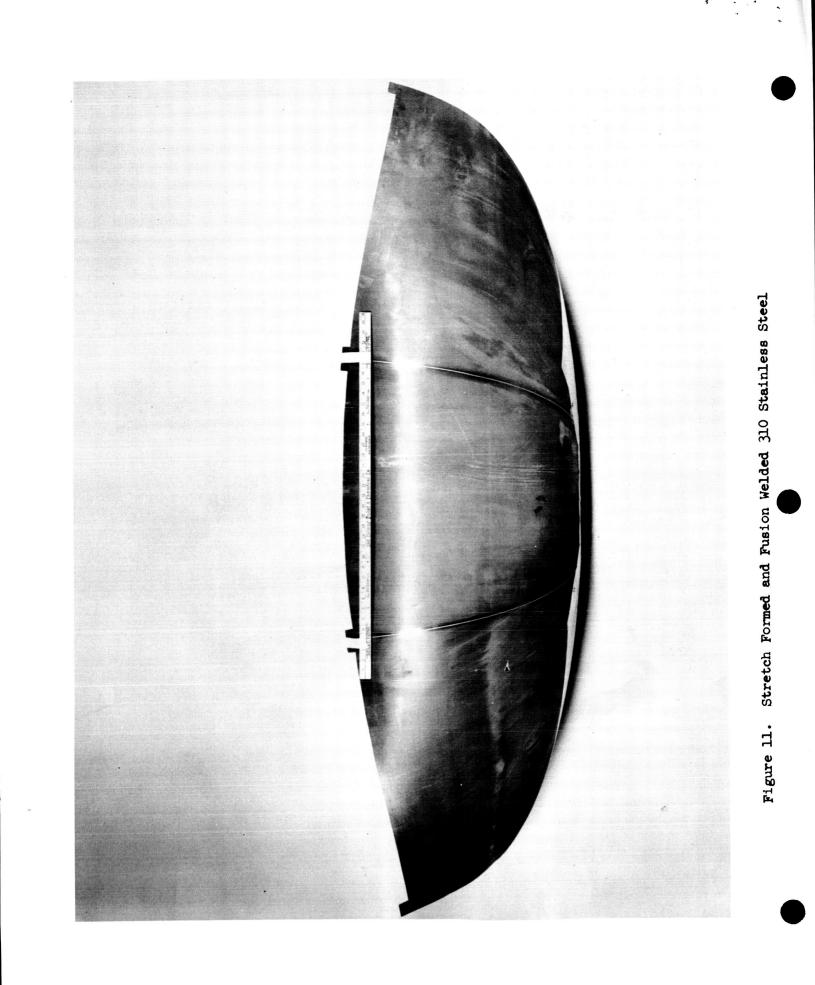


Figure 10. Examples of Cupping Test Specimens



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