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# Study of Cryogenic Storage **Tank Fatigue Life**

Low Temperature Mechanical Testing of AISI 304 and 310 Stainless Steels

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# Study of Cryogenic Storage Tank Fatigue Life

Low Temperature Mechanicol Testing af AISI 304 and 310 Stainless Steels

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STUDY OF CRYOGENIC STORAGE TANK FATIGUE LIFE Low Temperature Mechanical Testing of AISI 304 and 310 Stainless Steels R. P. Reed, R. L. Durcholz, R. E. Schramm, and T. J. Patrician

Two 300 series stainless steels were tested for impact-fatigue life and tensile properties as a function of temperature, percent transformed phase (martensite), stress level, and specimen geometry. These alloys were studied to predict the fracture characteristics of parent material and weld joints in large cryogenic dewars which are subjected to periodic stresses. Normally, AISI 304 is employed in the construction of such dewars. Under fatigue loading conditions at cryogenic temperatures, the behavior of AISI 304 is complicated by the gradual transformation to a martensitic structure. The influence of this transformation on the fracture characteristics was studied. For this purpose, a new impact-fatigue test and necessary equipment were developed.

The fatigue strength of AISI 304 and 310 exhibit similar trends, but at a given fatigue life and temperature, the fatigue strength of AISI 304 is slightly superior. AISI 310 displays an endurance limit of about 20,000 psi;\* the 304 limit is 25-30,000 psi. However, the lowest endurance limit (weakest) specimens appear to be the triaxiallyloaded AISI 304 weld specimens which have an endurance limit near 10,000 psi. Little temperature-dependence of the impact-fatigue properties was observed.

Key words: Fatigue; fracture; impact; low temperature; mechanical property equipment; stainless steel.

<sup>\*</sup> For ease in interpretation all stress values in the text are expressed in units of psi. Conversion to other stress units are  $10^{\circ}$  psi = 0.703 kg/mm<sup>2</sup> = 6.90 × 10<sup>6</sup> N/m<sup>2</sup>.

#### 1. Summary

The principal results of this report are briefly summarized below.

- The impact-fatigue strength of AISI 304 is superior or equal to the impact-fatigue strength of AISI 310 under all conditions in the temperature range 76 to 294K.
- 2. The endurance limit generally appears at about 10<sup>5</sup> cycles. At all temperatures (294, 195, 76K), the limit can be roughly approximated by using the room temperature tensile yield strength. Possible critical exceptions are under triaxial stresses at 195 and 76K, where the endurance limit is less than the room temperature yield stress. In these cases, a considerable extra safety factor in designing is desired.
- 3. Welds exhibit less strength under impact-loading conditions than the parent material: under uniaxial loading conditions, approximately a 10% reduction was found; under biaxial conditions about a 15% reduction was found. Although triaxial loading conditions could not be tested, on the basis of our results, it is expected that this configuration (weld, triaxially applied stress) at low temperature would prove the weakest link in any structure.
- Cold working (11%) at 76K produces a 35-50% increase in AISI 310 fatigue strength and a 40-55% increase in AISI 304 fatigue strength.
- 5. The transformation to martensite during repeated impact cycles has little deleterious effect on fatigue life. Under uniaxial and biaxial loading conditions, the transformation

is beneficial, increasing the fatigue strength.

- 6. In the case of AISI 304, there is a good possibility of monitoring the fatigue life by measurement of the amount of ferromagnetic phase (martensite) by simple magnetic techniques.
- 7. Two studies remain in order to insure the mechanical stability at cryogenic temperatures of AISI 300 series stainless steels, undergoing partial martensitic transformations. These are: the impact-fatigue strength in the temperature range 4 to 76K and the influence on martensitic transformations and mechanical stability of differences in weld techniques.

#### 2. Introduction

The objective of this study is to predict the life of AISI 300 series stainless steel parent material and weld joints when subjected to periodic large, sudden stresses in the temperature range 300 to 76K. At present, knowledge concerning the endurance life and effects of martensitic transformation on fatigue strength is not available. To enable safe transport of liquid oxygen in large quantities (400 gallons and up), such knowledge must be obtained, since most dewar systems are now fabricated with AISI 304 stainless steel. Actual conditions were simulated to provide the most useful information to dewar design.

Brittle failure, from analysis of accident investigations, is listed as a factor of primary importance in causing system failures in cryogenic equipment. The continual exposure of liquid oxygen tanks to high stresses (for example during the carrier on-deck delivery) poses a serious question of possible fatigue-transformation failure. Failure may occur for two reasons:

> (1) When normal 304 stainless steel is stressed above its yield strength at temperatures below about 200K, a partial crystallographic transformation from austenite (face-centered cubic structure) to martensite (body-centered cubic structure) occurs. There is an associated local volume expansion of 4% caused by the transformation. This local volume expansion promotes very high stress concentrations in material that is not ductile, such as weld areas.

(2) Normal fatigue failure, under periodic stress above the yield stress, also may occur. The endurance or fatigue limit of the combination of 304 steel, weld joint, and large stresses has never been tested; and to our knowledge, no similar circumstances have been duplicated in previous usage. Therefore, prediction of tank and safety is very speculative.

Various types of fatigue tests have been devised and are identified in three general categories.

1. tension-compression

2. bending

3. torsion

The test specimen is subjected to constraints which fall into one of two classes,

1. constant load, bending moment, or torque.

2. constant strain, bending deflection, or twist.

To keep test times to a reasonable duration, the repetition frequency is usually on the order of  $10^2 - 10^4$  cycles/min. High frequencies can result in an artifically short life due to sample heating. There are, however, indications<sup>1</sup> that a low repetition rate, without any heating problem, can also lead to a short life.

Many investigations<sup>2-13</sup> have shown that the strain rate to which a metal is subjected does affect the microstructure and this, in turn, is reflected in the mechanical properties. A review of many of these influences has recently been published.<sup>14</sup> Of course, there are actual situations which are approximated poorly or not at all by the <sup>1</sup> Superscripts indicate literature references listed at the end.

standard tests. Among these is the sudden impact loading or jolt delivered at a frequency of about one per hour or day, such as might happen during handling, transportation, or in the transfer of fluid through pipes.

To approximate better the actual environmental loading conditions, a new type of fatigue test was devised. This new test was performed on a commercial impact machine, modified to apply repeatedly the same impact load to the specimen (see Appendix). This report describes the tests carried out in the temperature range 76 to 297K, documents all test results, and discusses the significance of the data in terms of use in designing cryogenic equipment. 3. Test Materials, Specimen Preparation and Equipment

We tested two austenitic stainless steels, AISI 304 and 310; Table 1 gives the nominal composition of these alloys. AISI 310 is a crystallographically stable alloy with a face-centered cubic (fcc) structure, while annealed AISI 304 is known to undergo a martensitic phase transformation from fcc to hexagonal close-packed (hcp) and bodycentered cubic (bcc) structures. This transformation always occurs on deformation of the 304 steel below room temperature, and, depending on the exact composition, may occur spontaneously on cooling to the vicinity of 76 K.<sup>5</sup>

#### Table 1.

Nominal Composition of AISI 304 and 310 Stainless Steels

AISI		Comp	osition (%)	
Туре	С	Cr	Ni	Mn
304	0.08 max	18.0-20.0	8.00-11.00	2.00 max
310	0.25 max	24.0-26.0	19.0 -22.0	2.00 max

Specimens were tested in three configurations:

- 1. flat, smooth, (uniaxial).
- 2. flat, notched (biaxial).
- 3. round, notched (triaxial).

The notches cause the load to be broken down vectorially into longitudinal and transverse components. In this way the flat specimens are subjected to biaxial stresses while the round specimens see three-way or triaxial stresses. Dimensions are shown in Figure 1. The flat samples were cut from sheet parallel to the rolling direction, and the round specimens were produced from bar stock. By computing the stress concentration factor,  $K_t$ , from the formula



Figure 1. Sample geometries for tensile and impact tests: a. Uniaxial, thickness = 0.1 in; b. Biaxial, thickness = 0.1 in; c. Triaxial.

# $K_t = \sqrt{\frac{\frac{1}{2} \text{ distance between notches}}{\text{ notch radius}}}$

it has the value 4.2 and 4.5 for the biaxial and triaxial geometries, respectively. After machining, the flat samples were mechanically ground and polished. All samples were then vacuum annealed at 1000°C for one-half hour and air cooled. The majority of tests involved these materials as annealed, but some samples were subjected to further treatment to check the effect of chemical and physical variables on the impact fatigue life.

AISI 304 uniaxial and biaxial specimens, prepared from a melt which transformed spontaneously on cooling, were thermally cycled ten times between 297K and 76K. This produced about 2-7% martensitic products prior to the impact tests. Also, several 304 uniaxial specimens were held at 813K for three hours in air to test possible effects of sensitization, or the formation of intergranular carbides.

To get a quantitative idea of the influence of cold-work on impact-fatigue life, uniaxial specimens of both alloys were mounted on a tensile-testing machine and stressed at 76K to 11% strain; in the 304 this produced about 50% martensite. Those samples to be tested at 76K were maintained at that temperature until broken; the samples for the 297K tests were warmed just prior to use.

Welding introduces another very important variable. Welded plates were prepared from which uniaxial and biaxial samples were machined. Notches were ground both in the weld area and at two positions in the heat-affected zone. The technique was as follows:

material: AISI 304 stainless steel, 1/8 inch thick plate;

AISI 308 stainless steel weld rod, 0.035 inch diameter wire.

preparation: plates were machined, deburred, electro-

cleaned, rinsed, then electro polished. The caustic electrocleaning solution consisted of 8 oz. of Oakite<sup>\*</sup>#160 per gallon. This was run at 160°F(344K) at 6 volts. The plates were electro polished in 70% phosphoric acid, 30% sulfuric acid at 140°F (333K) using 9 volts for 4 minutes. Finally, the plates were again rinsed in water.

welding procedure: weld was butt-type in a flat position, using machine welding techniques. One pass, using an average welding speed of 18 inches per minute, was made over a length of 20 inches. A 0.09 inch gap was maintained by tacking 3.5 inch intervals. The plates were held down pneumatically and a Pandjiris weld positioner was employed.

welding atmospheres: a mixture of 30% He and 70% Ar

at an average flow of 10 cubic feet per hour was used. inspection and finishing: weld beads were inspected using standard radiographic techniques for porosity. Welds of poor quality were rejected. Weld beads were then machined off, the sample was polished, and magnetic permeability measurements made using a Magne-Gage. No further heat treatment was used prior to testing. The samples used in the microscopic analysis were of the uni-

<sup>\*</sup>Trademark. Precise specification of the material employed has been necessary to make the description of this apparatus sufficiently meaningful. Identification of this material or its manufacturer by the National Bureau of Standards in no way implies a recommendation or endorsement by the Bureau. Furthermore, use of other trade names in this paper is for the sake of clarity and does not in any way imply a recommendation or endorsement by the Bureau.

axial geometry, but thinner and with a wider reduced section (Fig. 2). They were annealed as noted for the impact specimens.

The impact-fatigue tests were conducted using a Riehle impact machine which had undergone major modifications; the details on the automation of this device and the cryogenic system used with it are presented in a paper to be published in Section C, Journal of Research, NBS (See Appendix). A standard Instron testing machine served for the tensile tests. The amount of ferromagnetic bcc martensite produced during these tests was recorded by measurements with a Magne-Gage, an instrument consisting of standard bar magnets affixed to torsion balance equipment.

The microscopic studies were done with a standard optical metallograph and electron microscope.



Figure 2. Electron microscopy specimen, thickness = 0.1 in.

#### 4. Measurements and Calculations

In the documentation and description of our data, a number of symbols and definitions are employed. The following discussion identifies these.

Definitions of Symbols Used

А	Sample area outside necking region after fracture.
A <sub>F</sub>	Sample area at fracture.
A <sub>0</sub>	Initial sample area.
E	Elastic modulus.
$E_{F}$	Absorbed energy density required to deform sample.
е	Elongation in l inch.
L	Sample load.
ł	Gauge length just before necking.
lf	Gauge length after fracture.
lo	Initial gauge length.
N	Number of impact cycles to fracture.
RA	Reduction in area before necking.
RAN	Reduction in area at neck.
Т	Temperature.
V	Sample volume after fracture.
Vo	Initial sample volume.
ė	Strain rate.
$e_{\rm E}$	Engineering strain.
€Ţ	True strain.
∩ <sub>A</sub>	Applied or engineering stress.
OF	Fracture stress.
$\sigma_{T}$	True stress.
au	Ultimate or tensile strength.
J	Yield strength (.002 offset).

Direct measurements are made on:

A,  $A_F$ ,  $A_0$ , L,  $\ell_F$ ,  $\ell_0$ , N, T, and  $\dot{\varepsilon}$ 

During the tensile tests, a strip chart recorder was used to continuously note the engineering strain,  $\epsilon_{\rm E}$ , as a function of the sample load, L. The engineering stress,  $\sigma_{\rm A}$ , is given by

$$\sigma_{A} = \frac{L}{A_{0}}$$

The stress-strain curve was then easily constructed from the strip chart record and used to determine the ultimate and yield stresses,  $\sigma_u$  and  $\sigma_y$ , and the elastic modulus, E. Figure 3 illustrates this graphically.



Figure 3. Determination of E,  $\sigma_{u}$ , and  $\sigma_{v}$  from a stress-strain curve.

Reductions of area and elongation are determined directly from sample measurements.

$$RA = \frac{A_0 - A}{A_0} = 1 - \frac{A}{A_0}$$
$$RAN = \frac{A_0 - A_F}{A_0} = 1 - \frac{A_F}{A_0}$$
$$e = \frac{l_F - l_0}{l_0}$$

In the notched samples, both biaxial and triaxial, little, if any, necking occurs so  $A = A_F$  and RA = RAN.

Fracture stress is

$$\sigma_{\rm F} = \frac{\rm L}{\rm A_{\rm F}}$$

For the tensile tests this is most easily determined from

$$\sigma_{\rm F} = \frac{\sigma_{\rm u}}{1 - \rm RAN}$$

While it is true that dislocations and vacancies are introduced into the crystalline structure during deformation, it is nearly true that the volume of the sample remains constant, i.e.

So

Since:

it follows that 
$$1 + \varepsilon_{\varepsilon} = \frac{1}{1 - RA}$$
,

and

$$=\frac{RA}{1-RA}$$

€E

From this the true strain can be determined.

$$\epsilon_{\tau} = ln\left(\frac{l}{l_{0}}\right) = ln\left(1 + \epsilon_{E}\right)$$

The true stress is

$$\sigma_{T} = \frac{L}{A}$$

Again assuming constant volume:

$$\sigma_{T} = \frac{L}{A_{0}} - \frac{A_{0}}{A} = \sigma_{A} - \frac{\ell}{\ell_{0}} = \sigma_{A}(1 + \epsilon_{E})$$

There are basically two stages in the fatigue life of a sample:

1. Uniform strain.

2. Necking followed by fracture.

Most of the energy is absorbed during the uniform strain portion. Once necking has begun, almost all further energy is concentrated in that small volume and fracture quickly follows. To make calculation possible, it is assumed that the only significant absorbed energy is that required to just bring the sample to the point of necking. The density of absorbed energy required to deform the sample is then:

$$E_{F} = \frac{L(\ell - \ell_{\circ})}{V} = \frac{L(\ell - \ell_{\circ})}{V_{\circ}} = \frac{L}{A_{\circ}} \quad \frac{\ell - \ell_{\circ}}{\ell_{\circ}},$$

$$E_F = \sigma_A \varepsilon_E$$

#### 5. Experimental Results

#### 5.1 Mechanical--Impact

The impact-fatigue data for AISI 304 and 310 stainless steels are presented in Tables 2 and 3 and are plotted as S-N curves (applied stress vs. number of cycles) in Figures 4-11. The fatigue life curves in Figures 12-14 give a comparison of these two alloys for durations of  $10^2$ ,  $10^3$ , and  $10^4$  cycles. Some results are immediately apparent from these curves:

 A decrease in temperature increases the impact-fatigue life.
 Fatigue life of uniaxially stressed specimens is generally longest, followed by biaxially stressed specimens. Specimens stressed under triaxial conditions are usually weakest.

(3) In the uniaxial configuration, the fatigue strength of 304 is superior to that of 310. At room temperature, the difference is about 5-15% and at 76K it is 25-30%. In Figures 7 and 8, the data for the 304 samples that were sensitized or precycled to 76K fall in line with the annealed points, indicating very little or no effect on the impact-fatigue behavior by these thermal treatments. This agrees with previous work on sensitized 304 subjected to high strain rates.<sup>23</sup>

(4) The shape of the curves in Figures 10 and 11 for the 304 weldments exhibit the same general behavior as the annealed material. In the uniaxial geometry, the strength is slightly reduced by welding; however, this decrease depends on temperature - about 20% at 297K and 8% at 76K for a life of  $10^{\circ}$  cycles. The results are similar for a  $10^{4}$  cycle life (Figure 14). The strength of biaxial samples is nearly unchanged at room temperature but reduced at 76K. The most remarkable thing here is the insensitivity of fatigue life to the relative position of weld and notch. In Figure 11, a single curve at each temperature could be drawn through the data points for all three notch positions.

E	t	Table 2.	. Impact-F	atigue Re	sults, A	DAN	C.	Q	Ь
	(10 <sup>3</sup> psi)*	$(10^3 \text{ psi})^*$	Z	e (%)	KA (%)	KAN (%)	$\epsilon_{\rm E}$	ε <sub>τ</sub> (%)	(10 <sup>3</sup> ft-lb/in <sup>3</sup> )*
			Uniaz	xial, Ann€	ealed				
2	70°0	217.2	6, 346	41	21	68	27	24	1.6
2	76.1	233,3	4,097	44	23	67	30	26	1。9
2	112。9	297.7	57	65	33	62	49	40	4.6
2	150,5	368.4	12	54	29	59	41	34	5.1
2	187。2	500°0	2	54	27	62	37	31	5 <sub>°</sub> 8
2	90°3	280.0	386	60	32	68	47	39	ъ ъ С
2	114.1	295.7	51	51	28	61	39	33	3°7
ŝ	159.1	378。3	13	45	24	58	32	28	4.2
9	87.0	229°8	20,844	50	25	62	33	29	2.4
9	112.3	323.0	1, 298	57	28	65	39	33	3.6
9	151.4	350.0	84	61	30	57	43	36	5.4
9	188。2	406。9	18	47	31	54	45	37	7.1
			Biax	ial, Anne	aled				
2	53 <b>.</b> 8	81.0	4,897		34		52	42	2°3
2	60.5	86.8	3, 809		30		43	36	2.2
2	75.7	137.3	501		45		82	60	5.2
2	112°3	205.9	∞		45		82	60	7°2
2	74。5	117.6	869		37		59	46	3.7
5	100.0	154.1	39		35		54	43	4.5
S	113.5	187.5	6		39		64	49	6 <b>.</b> 1
S	201.1	327.1	1		39		64	49	10.7

2.0	1.6	2.7	3,5		0.3	0,8	6°0	2,3		0.03	0, 1	0.9	0,1	0.6	0.8		0.7+	1.4	6.9	7.3	5. 3	5.8		
28	20	25	29		6	17	17	30		1	2	11	2	8	6		$10^{+}$	17	36	30	33	29		
32	22	28	34		6	18	18	35	-	-	2	12	2	8	6		$11^+$	18	43	35	39	33		
																		59	54	58	40	45		
24	18	22	28	ealed	8	15	15	26	ŗ	٦	2	11	2	7	8	1% at 76 K	$10^{+}$	15	30	26	28	25		
				ıxial, Ann												Strained 1	23+	42	37	34	62	61	/mm <sup>3</sup>	
4,992	1,892	30	9	Tria	2,138	889	645	14		144 °C	1,684	429	2,518	238	33	Uniaxial,	61,300	3,087	2	4	1, 5,84	51	/mm <sup>2</sup> 828 ioules	2 4 5 7 1
6°26	115.9	145°8	212。1		43 <b>。</b> 4	61.8	67.7	105.9	20 2	, or	53.5	98°6	54°0	89.8	120.7			229.9	388 <b>。</b> 0	545.5	271.4	383.3	si = 0.703 kg -lb/in <sup>3</sup> = 0.0	ured at grip
74。4	89.8	114。1	153.0		40 °1	52.5	57.4	78.8	00000	0.00	52.6	87.7	53°0	83。9	111.5		78.7	93.3	193.9	251.4	164.2	211.8	$* 10^3 \text{ p}$ $10^3 \text{ ft}$	+ Fract
76	7ó	76	76		297	297	297	297	105	17.0	195	195	76	76	76		297	297	297	297	76	76		

	E <sub>f</sub> 10 <sup>3</sup> ft-lb/in <sup>3</sup> )*		2.3+	4.8	4.5	5.0	8.5	5.9	5.9	6.7	8.8	1.3+	3.4	1.7	1.0 <sup>+</sup>	2.5	3.9	7.1		1.5	2.7	6.0	
	ε <sub>1</sub> (%) (		33+	49	54	44	60	42	51	44	48	15+.	24	14	$10^{+}$	20	24	28		28	43	67	
304	Е Е ( %)		39+	64	72	56	82	52	67	56	61	$16^{+}$	27	15	$11^{+}$	22	27	32		32	54	96	
TCTY 'S	RAN (%)			59	60	68	67	67	65	62	66		61	60		52	45	47					
Results	RA (%)	aled	28+	39	42	36	45	34	40	36	78	$14^{+}$	21	13	$10^{+}$	18	21	24	aled	24	35	49	
רוד מרוצתם	e (%)	ial, Annea	42+	39	26	71	114	65	100	7 0	68	$18^{+}$	42	40	14 +	32	35	38	ial, Annea				
on minha	Z	Uniaxi	24,500	10, 340	9,300	930	100	54	45	14	6	12,000	35	23	10,270	1,548	435	19	Biaxi	4,061	975	58	
J 4 2 3 4	0F (10 <sup>3</sup> psi)*			224.0	189.1	329.0	379.2	411.7	304.3	378.3	500.0		383.5	335.2		286.0	315.3	500.0		72.1	90.3	147.3	
:	(10 <sup>3</sup> psi)*		71.8	90.8	75.3	106.6	123.8	137.3	106.1	143.6	172.4	100.9	148.7	135.2	111.1	137.9	173.3	266.7		54.4	59.6	75.3	
	Т (К)		297	297	297	297	297	297	297	297	297	195	195	195	76	76	76	76		297	297	297	

Table 3. Impact-Fatigue Results, AISI 3

0°8	3, 2	4,6	7°2	0°0	0.3	0.4	1.4	1.7	2.9	<b>4.</b> 0		0.5	0.9	8°5	0.2	0,1	3.6	0,2	0.4	1。8
11	33	44	46	6	4	9	15	17	22	24		14	17	67	5	2	30	4	6	15
12	39	56	59	6	4	9	16	19	25	27		15	18	96	Ŋ	2	35	4	,0	16
11	28	36	37	8	4	9	14	16	20	21	l, Annealed	13	15	49	£	2	26	4	- ,0	14
2,615	676	128	17	4,149	2,073	1,227	569	132	57	6	T riaxial	5,568	955	13	3 <b>,</b> 454	1,231	644	3.156	458	72
91.1	136.8	189。2	250°0	82.8	86.0	91°3	121。7	128.8	172.8	222。9		50, 1	68°0	208°8	55°5	71。3	120°0	55, 1	92.7	156.1
81.5	98°4	120.7	156.4	76.3	82°3 ·	85°7	104.3	108.2	138.6	176.8		43 <b>。</b> 5	58°0	105.9	52°9	70.1	89°2	53, 0	87.5	135,0
195	195	195	195	76	76	76	76	76	76	76		297	297	297	195	195	195	76	76	76

	E <sub>F</sub> (10 <sup>3</sup> ft-lb/in <sup>3</sup> )*		2°5	4°9	2°6	4°4		0.6	2°3	4.7	5, 1	7.8	3°9		5°0	6 <b>.</b> 1	1°6+	4°.3
ued)	$\varepsilon_{\uparrow}$ (%)		29	36	20	21		17	39	56	50	64	26		54	49	14	24
contin	$\epsilon_{\rm E}$ ( $^{0\!/_{\rm O}}$ )		33	43	22	23		18	47	75	64	89	30		72	ó4	15	27
ISI 304 (	RAN (%)	M	99	65	51	34								لتر ه	54	73		40
sults, A	RA (%)	ed to 76 I	25	30	18	19	d to 76 k	15	32	43	39	47	23	l at 1000	42	39	13+	21
atigue Re	e (%)	Precycle	44	61	28	32	Precycle							ensitized	80	73	$18^+$	32
• Impact-F	Z	Uniaxial,	491	16	7,521	15	Biaxial,	10,340	1,279	109	16	247	06	Uniaxial, S	10,506	38	11,255	104
Table 3	$\sigma_{\rm f}$ (10 <sup>3</sup> psi)*		262.5	388.8	286。0	347。1		50.0	86。8	130.8	155.6	198.1	207。4		183.3	428.5		315.3
	$(10^3 \text{ psi})*$		89°8	137°9	139。3	228。1		42.6	58°3	74。9	94。8	105.0	157.3		83.7	114.1	131.5	190.2
	Т (К)		297	297	76	76		297	297	297	297	76	76		297	297	76	76

				0°8 2°0 8	1.1 2.6 7.6
				28 46 51	20 30 48
				22 59 67	22 35 61
Uniaxial, Welded	47 53 65	5 5 5 4 7 4	26 48 52	at Weld) 18 37 40	18 26 38
	35 36 57	44 55 46	34 46 47	Welded (Notch	
	11, 518 67 31	8,818 1,780 33 7	9,200 584 20	Biaxial, 6,446 1,137 11	3, 888 269 10
	114.2 215.4 323.0	205.0 222.7 353.6 406.9	153。3 291。0 388。0	54.9 94.1 150.0	72.3 120.9 241.4
	60.5 90.3 112.9	84.6 105.3 127.3 189.2	114。1 151。3 188。1	44.9 59.3 89.8	59.6 89.8 149.7
	297 297 297	195 195 195	76 76 76	297 297 297	76 76 76

	$E_{\rm F}$ (10 <sup>3</sup> ft-lb/in <sup>3</sup> )*		1°3	<b>4</b> °3	2.2	10.2		1.5	3°8	0.6	4 <b>.</b> 8		1.2	$1.4^{+}$	3.1	1.9	2.1	3.2		
ue Results, AISI 304 (continued)	ε <sub>τ</sub> (%)	n Weld) 30 26	26	52	33	90		30	47	10	36		11	$10^{+}$	13	10	10	10		
	€Е (%)		30	69	39	39 82 82 81 61	10	43		12 10 <sup>+</sup> 14 11 10										
	RAN (%)		23		28		n Weld)	26	38			K	30		52	47	47	30		
	RA (%)	16" fro		41		45	/8" fror			6	30	% at 76	11	+6	12	10	6	10		
	e ( %)	(Notch 1/					l (Notch 1					trained 11	29	$25^{+}$	40	34	37	33	./mm <sup>3</sup>	
npact-Fatig	N	ial, Welded	3, 184	92	2,620	D	cial, Weldec	2,435	26	2,875	∞	Uniaxial, St	6,844	128	10	1,456	177	10	kg/mm <sup>2</sup> ). 0828 joule	ip .
Table 3. In	σ <sub>F</sub> (10 <sup>3</sup> psi)*	Biaxi	67.6	127。3	94.7	274。5	Biax	70.5	120°7	74。1	193.8		172.3		552.6	381.8	482.8	495.6	psi = 0.703 ft-lb/in <sup>3</sup> = 0	ctured at gr
	σ <sub>A</sub> (10 <sup>3</sup> psi)*		52.1	75.3	67°7	149。7		52,1	75.3	67.7	134。8		120.4	170.7	265.8	202.4	256.1	345.7	$* 10^{3}$ 10 <sup>3</sup>	+ Fra
	Т (К)		297	297	76	76		297	297	76	76		297	297	297	76	76	76		



Figure 5. S-N curve for AISI 310, biaxial.





Figure 9. S-N curve for AISI 304, triaxial.






Figure 13. Fatigue life 10<sup>3</sup> cycles.



Figure 14. Fatigue life at 10<sup>4</sup> cycles.

Figure 15 allows comparison with some previous fatigue life measurements<sup>15-19</sup> that were conducted in tension-compression. While the impact and conventional fatigue curves do have the same general structure, there are some differences:

(1) The impact S-N curves fall at a higher stress level.

(2) The fatigue limit on impact, uniaxial stress appears to occur at or above the yield strength (0.002 offset) as determined from tensile tests; in conventional fatigue the limit is well below this level.

These factors are probably related to the increase in yield strength with increasing strain rate.

Data from the literature  $^{20-22}$  show that cold-work has a significant effect on the mechanical properties of 18 Cr-8 Ni steels, and Figures 15 and 16 show this effect on fatigue life. To make a controlled



Figure 15. Comparison of impact-fatigue data for annealed AISI 304 with that obtained in tests using tension-compression at low strain rates (Refs. 15-19). The triaxial samples of Krempl<sup>17</sup> had a stress concentration factor,  $K_{\star}$ , of 3.3.



Figure 16. Fatigue-life data (Ref. 20-22) for cold-worked (to 210,000 psi tensile strength) AISI 304. Tests were conducted by tension-compression at low strain rates. The biaxial samples had a stress concentration factor,  $K_t$ , of 2.75 to 3.9.

check of the effect of cold-work on 304 and 310, uniaxial specimens were strained 11%, as noted above, and then impacted in the usual manner. Figure 17 shows the results and comparison with Figures 4 and 7 indicates a considerable strengthening at both 297 and 76K. At a life of  $10^3$  cycles, the improvement in the applied stress is about 40-50%. Also, the superiority of AISI 304 over 310 is again evident.

Figures 18-25 show that the fracture stress for all materials and all configurations follows a pattern very similar to the applied stress with two exceptions:

(1) The 310 biaxial data (Figure 19) lie on a single curve for all temperatures.

(2) The 304 biaxial data (Figure 22) show a small separation between the precycled and annealed room temperature curves.



Figure 17. Impact-fatigue data for cold-worked uniaxial specimens. The 304 contained 50% martensite after straining.



Figure 18. Fracture stress for AISI 310, uniaxial.







Figure 20. Fracture stress for AISI 310, triaxial.



Figure 21. Fracture stress for AISI 304, uniaxial.



Figure 22. Fracture stress for AISI 304, biaxial.



Figure 23. Fracture stress for AISI 304, triaxial.



Figure 24. Fracture stress for AISI 304, uniaxial, welded.



Figure 25. Fracture stress for AISI 304, biaxial, welded.

Another technique to characterize the mechanical behavior is to consider the applied energy needed to fracture specimens. This may be considered, to a first approximation, as the product of the applied stress and specimen strain (A more detailed approximation is described in Section 4 and used in all calculations). This computed deformation energy density,  $E_F$ , is plotted versus the log of the number of cycles to failure, N, in Figures 26-32. In the range tested, these curves can be approximated by an exponential relation,

$$N = N_0 \exp\left(\frac{-mE_f}{kT}\right)$$

between the number of cycles to failure, N, and  $E_F$ , where N<sub>0</sub> and m are material constants for a given stress configuration and k is Boltzmann's constant. In all but one case, the amount of energy that must be absorbed by the sample before fracturing decreases with the temperature. The one exception to this decrease in absorbed energy with temperature is the AISI 310 uniaxial (Figure 26). Figures 29 and 30 indicate that the presence of martensite in AISI 304 prior to testing decreases the energy absorbed before fracture. The relative position of weld and notch has very little effect on absorbed energy at 297K, but does exert a considerable influence at 76K (Figure 32). Figure 33 displays summary curves at 10<sup>3</sup> cycles and the deviation of the AISI 310 uniaxial is obvious.

The ductile behavior of the two steels can be seen in Figures 34 and 35. Again, the room temperature differences are small but increase considerably as the temperature decreases. The low ductility of AISI 304 at 76K is probably due to the large amount of martensite present.



Figure 26. Deformation energy density for fracture of AISI 310, uniaxial.



Figure 27. Deformation energy density for fracture of AISI 310, biaxial.



Figure 28. Deformation energy density for fracture of AISI 310, triaxial.



Figure 29. Deformation energy density for fracture of AISI 304, uniaxial.



Figure 31. Deformation energy density for fracture of AISI 304, triaxial.



Figure 33. Summary of deformation energy density for fracture at 10<sup>3</sup> cycles.



Figure 35. Uniform reduction of area for fracture at  $10^3$  cycles.

The progress of one impact test on a uniaxial 304 specimen at 297K was interrupted frequently to make measurements on the elongation, reduction of area, and amount of martensite formed. From the results in Figure 36, it appears that all three quantities reach a plateau and then increase abruptly before fracture.

## 5.2 Mechanical -- Tensile

The results of the tensile tests on both steels are presented in Table 4. The sample elongation shows that the ductility does decrease as the strain rate increases. The considerable increase in yield strength noted for the moderate strain rate of 20 min<sup>-1</sup> may possibly be due to a response time in the strip chart recorder too low to properly follow the load extension curve at this higher crosshead speed.

The inverse relationship between strain rate and ductility has been observed previously.<sup>9,11,23</sup> An increasing strain rate has also



Figure 36. Parameters during an impact-fatigue test of AISI 304 at 297 K.

Table 4. Tensile Results.

MISI TypeConfigurationTT $_{0}^{-1}(1,00^{2},psi)*$ $_{0}^{-1}(1,0^{2},psi)*$ $_{0}^{-1}(1,0^{2},psi)*$ $_{0}^{-1}(1,0^{2},psi)*$ $_{0}^{-1}(1,0^{2},psi)$ $_{0}^{-1}(1,$
AISI Type         Configuration (K)         T (K)         T (min <sup>-1</sup> ) $(10^3 \text{ psi})$ (10 <sup>3</sup> psi) $(10^3 \text{ psi})$ (
AISI Type         Configuration (K)         T $\frac{e}{(K)}$ $\frac{o_1}{(M)}$ $\frac{o_1}{(M)}$ $\frac{o_1}{(M)}$ $\frac{o_1}{(M)}$ $\frac{e}{(M)}$ $\frac{R}{(M)}$ RAN           7.1ype         (K)         (K)         (m) $\frac{o_1}{(M)}$
AISI Type         Configuration mixed         T $\frac{e}{(X)}$ $\alpha_{\gamma}(.002)$ $\alpha_{\gamma}(.010)$
AISI TypeConfigurationT T (K) $^{\circ}_{(1,0)}$ $^{\circ}_$
AISI TypeConfigurationT (K)T (min-1) $a_{i}$ </td
AISI TypeConfigurationT (K)T (min-1) $\sigma_{1}$ (103 psi)# $\sigma_{1}$ (103 psi) $\sigma_{1}$ (103 psi)# $\sigma_{1}$ (103 psi) $\sigma_{1}$ (103 psi) $\sigma_{1}$ $\sigma_{1}$ (103 psi) $\sigma_{1}$ (103 psi) $\sigma_{1}$ $\sigma_{1}$ (103 psi) $\sigma_{1}$
AISI TypeConfigurationT (K) $\overset{\circ}{(mni-1)}$ $\overset{\circ}{(10^3 \text{ psi})}$ $\overset{\circ}{(10^3 \text{ psi})}$ Type(K)(min-1) $(10^3 \text{ psi})$ $(10^3 \text{ psi})$ $\overset{\circ}{(10^3 \text{ psi})}$ 304uniaxial $297$ $0.002$ $38.9$ $100.0$ 304uniaxial $297$ $0.22$ $38.9$ $92.5$ 304uniaxial $297$ $0.22$ $38.9$ $92.5$ 304uniaxial $76$ $0.22$ $38.9$ $94.7$ $304$ uniaxial $76$ $0.22$ $38.9$ $99.3$ $304$ uniaxial $76$ $0.22$ $38.0$ $93.3$ $304$ uniaxial $76$ $0.22$ $38.0$ $93.3$ $304$ uniaxial $76$ $20.2$ $1102.74$ $193.6$ $310$ uniaxial $76$ $20.2$ $38.0$ $93.3$ $310$ uniaxial $76$ $20.2$ $38.0$ $93.3$ $310$ uniaxial $76$ $0.002$ $38.0$ $99.2$ $310$ uniaxial $76$ $0.022$ $38.0$ $99.4$ $310$ uniaxial $76$ $0.02$ $38.0$ $99.2$ $310$ uniaxial $76$ $0.022$ $84.4$ $178.2$ $310$ uniaxial $76$ $0.022$ $88.4$ $178.3$ $310$ uniaxial $76$ $0.022$ $84.4$ $178.5$ $304$ uniaxial $76$ $0.022$ $49.5$ $85.8^{\ddagger}$ $304$ biaxial $195$ $0.022$
AISI TypeConfigurationT (K) $\stackrel{\circ}{(min^{-1})}$ $\stackrel{\circ}{(10^3 psi)*}$ Type(K)(min^{-1}) $(10^3 psi)*$ 304uniaxial $297$ $0.002$ $38.9$ 304uniaxial $297$ $0.022$ $38.9$ 304uniaxial $297$ $0.022$ $38.9$ 304uniaxial $76$ $0.022$ $38.9$ 304uniaxial $76$ $0.002$ $38.6$ 304uniaxial $76$ $0.022$ $38.0$ 304uniaxial $76$ $0.022$ $38.0$ 310uniaxial $76$ $20.2$ $119.5^{+}$ 310uniaxial $76$ $20.2$ $38.0$ 310uniaxial $76$ $0.022$ $38.0$ 311uniaxial $76$ $0.022$ $38.0$ 310uniaxial $76$ $0.022$ $38.0$ 304uniaxial $76$ $0.022$ $49.5$ 304biaxial $76$ $0.022$ $49.5$ 304uniaxial $76$ $0.022$ $49.5$ 304biaxial $76$ $0.022$ $49.5$ 304biaxial $76$ $0.022$ $49.5$ 305biaxial $76$ $0.022$ $49.5$ 304biaxial
AISI TypeConfigurationT (T)T (min-1)Type 304uniaxial $297$ $0.002$ 304uniaxial $297$ $0.002$ 304uniaxial $297$ $0.002$ 304uniaxial $766$ $0.202$ 304uniaxial $766$ $0.202$ 304uniaxial $766$ $0.202$ 304uniaxial $766$ $0.202$ 310uniaxial $766$ $0.002$ 310uniaxial $766$ $0.002$ 310uniaxial $766$ $0.002$ 310uniaxial $766$ $0.022$ 304uniaxial $766$ $0.002$ 303uniaxial $766$ $0.022$ 304uniaxial $766$ $0.022$ 304uniaxial $766$ $0.022$ 303uniaxial $766$ $0.022$ 304biaxial $766$ $0.022$ 304biaxial $766$ $0.022$ 304biaxial $766$ $0.022$ 304biaxial $766$ $0.022$ 303biaxial $766$ $0.022$ 310biaxial $766$ $0.022$ 310biaxial $766$ $0.022$ 311biaxial $766$ $0.022$ 310biaxial $766$ </td
AISI TypeConfigurationT (K)TypeType(K) $Type$ uniaxial297 $304$ uniaxial297 $304$ uniaxial297 $304$ uniaxial76 $304$ uniaxial76 $304$ uniaxial76 $304$ uniaxial76 $310$ uniaxial297 $310$ uniaxial297 $310$ uniaxial297 $310$ uniaxial76 $310$ uniaxial76 $304$ uniaxial76 $304$ biaxial76 $304$ biaxial297 $304$ biaxial297 $310$ biaxial297 $310$ biaxial297 $304$ biaxial297 $310$ biaxial76
AISI TypeConfigurationType304umiaxial304umiaxial304304umiaxial304304umiaxial304304umiaxial310310umiaxial310310umiaxial310310umiaxial310310umiaxial310310umiaxial310310umiaxial310310umiaxial310310umiaxial304304biaxial304303biaxial304310biaxial310310biaxial310310biaxial310310biaxial310310biaxial310310biaxial310310biaxial310310biaxial310310biaxial310310biaxial310310biaxial310
AISI Type 304 304 304 304 304 304 310 310 310 310 310 310 310 310 310 310

\* 10<sup>3</sup> psi = 0,708 kg/mm<sup>2</sup>
† These values are perhaps too high, see text for further discussion.
\* Fractured in weld.

been shown to decrease the amount of material transformed in metastable alloys.<sup>3,9,24,25</sup> In turn, the amount of martensite has been related directly to the tensile and yield strengths of the metal; generally they increase together.<sup>26</sup> As would be expected from these two observations, the tensile strength will tend to decrease as the strain rate is increased.<sup>9</sup>

True stress-true strain curves for both alloys have been recorded in Figures 37 and 38. These were constructed from the loadextension curve obtained during the tests, with the assumption that the elongation is equal to the engineering strain; this assumption should be valid until necking occurs shortly before fracture. The spread of some representative tensile test data<sup>5</sup>, 27-29 is shown in Figures 39 and 40, and the present data show general agreement with these results with the exception of the yield strength at 20 min<sup>-1</sup> as noted above.

## 5.3 Transformation Products in AISI 304

The amount of martensite transformation product formed in 304 is a function of strain, strain rate, temperature, and chemical composition. The amount of bcc phase present is most easily determined by measuring relative magnetic permeability. This can be performed quite rapidly with a Magne-Gage, a device which measures the force required to pull a standard magnet from the surface of the sample. Using this technique, careful observations have been made previously on transformed martensite during the progress of tensile tests.<sup>5</sup> In a similar manner, as noted above, we recorded measurements for an AISI 304 annealed, uniaxial sample during its impactfatigue life at 297K. Figure 41 indicates that, as in tensile tests, the percent martensite increases with the strain. In this same figure, point data are included from measurements on samples after being impacted to fracture. In general, the results at room temperature



Figure 37. True stress-true strain curves for tensile tested AISI 310.



Figure 38. True stress-true strain curves for tensile tested AISI 304.







Figure 41. Formation of martensite during tensile and impact tests.

seem to be much more dependent on alloy composition than they are at 76K.

In all of the 304 samples, the martensite fraction was recorded after fracture. The results in Figure 42 indicate that this value falls within rather narrow limits for each temperature and geometry.

# 5.4 Microscopic Observations

Tensile specimens of 304 and 310 stainless steels, both impact and slowly loaded (0.002 min<sup>-1</sup>), were examined using techniques of optical and transmission electron microscopy. Specimens were deformed in tension at either 76 or 297K to produce strains of approximately 3% or 9%. After a specimen was strained, it was examined using optical light microscopy to determine the nature of the slip traces resulting from the deformation. Following this, the specimen was subsequently chemically thinned and then electropolished to produce



Figure 42. Temperature dependence of martensite formed after impact-fatigue fracture in AISI 304.

foils suitable for examination in the electron microscope.

Examination of the tensile specimens, using optical microscopy, revealed the grain size and slip traces. When a grain is deformed, slip will occur initially on the slip system where the resolved shear stress exceeds the critical resolved shear stress,  $\tau_c$ . In facecentered cubic metals and alloys, there are 12 possible slip systems, defined by the four  $\{111\}$  slip planes which each contain three  $\langle 110 \rangle$ slip directions. In polycrystalline specimens, the primary slip system (the slip system on which  $\tau_c$  is first exceeded) is dependent on the orientation of a grain in respect to the applied load; therefore, for grains of different orientations, slip will produce slip traces in different directions relative to one another, delineating individual grains. Furthermore, initiation of conjugate slip, slip on a second slip system, is dependent on the orientation of a grain. Figure 43 shows an area of a specimen that was strained  $\sim 3\%$  where there are examples of grains having slip on the primary slip system only (grain A) and grains showing cross slip (grain B), slip on both the primary and conjugate slip systems (this is representative of both 304 and 310). The frequency of grains containing cross slip is an indication of the amount of plastic deformation in a specimen, since a random distribution of orientations for the grains in each specimen can be assumed. For both alloys, it was observed that there were substantially more cross slipped grains at 9% strain than at 3% strain and that the rate of loading did not affect the occurence of cross slip. The temperature of deformation likewise did not affect the frequency of observed cross slip. This is unexpected since T<sub>c</sub> is temperature dependent. It, therefore, appears that the application of the tensile load, either impact or slow, and the temperature of deformation had little influence on the slip behavior of these alloys.

Face-centered cubic alloys undergo three distinct stages of deformation characterized by three dislocation structures. These are as follows:

Stage I: low strain

(a) Long straight dislocations on the primary slip system.

(b) A few dislocation interactions, the beginning of conjugate slip.

(c) More dislocation interactions.

(d) Dislocation density at end of Stage I  $\sim 10^9$  cm<sup>-2</sup>.

Stage II: intermediate strain

(a) Rapid appearance of rough dislocation cell structure; not possible to distinguish individual slip planes.

(b) High density of loops in cell walls.

(c) Dislocation density at end of Stage II  $\sim 10^{11}$  cm<sup>-2</sup>.

Stage III: high strain to rupture

(a) Misorientation between cells increases.

(b) Boundaries of cells show a tendency to lie along primary or conjugate slip traces.

Evidence of Stage I is observed for both 304 and 310 stainless steel independent of deformation temperature (the deformation of 304 is complicated by a stress-induced martensitic reaction which will be discussed later). Figure 44 shows an example of deformation characteristics of the later part of Stage I for 310 stainless steel, which corresponds to  $\sim 3\%$  strain. The figure shows, as indicated by the arrows, the existence of two active slip systems, as well as dislocations which have interacted after piling up at (a) where two slip planes intersect; at (b) there is a stacking fault separating two partial dislocations. For  $\sim 9\%$ strain in the same alloy, which corresponds to early Stage II, a typical dislocation structure is given in Figure 45, which shows the initiation



Figure 43. 304 specimen strained  $\sim 3\%$ ; "A" grain with slip on primary slip system only, "B" grain showing slip on two systems. 200X

Then I win

Figure 44. 310 stainless steel strained  $\sim 3\%$ . Arrows indicate slip systems; (a) are dislocation interactions at dislocation pile-up where two slip planes intersect and (b) is a stacking fault separating two partial dislocations. 40,000X of a cell structure and a substantially higher dislocation density. The dislocation structures of 310 seemed to be the same, independent of loading and temperature; whereas the 304, while having the same general dislocation arrangements as the 310 at a corresponding strain, contained deformation twins in some grains when impacted to  $\sim 9\%$  strain (Figure 46).

Furthermore, since 304 stainless steel undergoes a stress induced martensitic transformation, the 304 specimens both impacted and slowly strained  $\sim 9\%$  at 297K, and all specimens strained at 76K contained hexagonal close-packed,  $\varepsilon$ , and body-centered cubic, a', phases associated with the transformation. Figure 47 shows an electron micrograph (a) and its selected area diffraction pattern (b) for a 304 stainless steel which has transformed after deformation. The diffraction pattern contains spots corresponding to the y, the parent facecentered cubic phase,  $\varepsilon$ , and a'. The long parallel bands in the micrograph are  $\varepsilon$  and the dark lenticular rods in the  $\varepsilon$  are the crosssections of a lathes. In this micrograph it is evident that a has transformed on two different variants, (a) and (b), of its habit plane. At low strain and 76K,there is evidence that  $^{\circ}$  is not always associated with the a, as can be seen in Figure 48. When subjected to high strains at room temperature or even low strains at 76K, independent of the method of loading, 304 stainless steel undergoes a martensitic transformation producing  $\alpha'$  and  $\varepsilon$ , which is associated with dislocation structures similar to those of 310 under similar conditions of deformation.

Optical microscopy observations of fractured specimens revealed that, in general, the fracture path is random, following neither grain boundaries nor planar (martensitic) features within the grains. Other microcracks were not detected. Since all specimens exhibited



Figure 45. Cell dislocation structure of 310 stainless steel after strain of  $\sim 9\%$ . 40,000X



Figure 46. Deformation twin in 304 stainless steel after ~9% strain. 100,000X



Figure 47. 304 stainless steel strained  $\sim 9\%$ . (a) Electron micrograph at 100,000X; (b) selected area diffraction,  $\varepsilon$  (hcp),  $\alpha$  '(bcc), and  $\gamma$ (fcc).



Figure 48. 304 stainless steel strained  $\sim 3\%$  at 76 K;  $\alpha'$  and  $\gamma$  present but no  $\epsilon$ . 16,000X

some necking prior to fracture, it is not surprising that other cracks were not observed; the necking served to concentrate the stress within a very limited region of the specimen.

Table 5 summarizes the deformation structures and the phases present for the alloys investigated under different conditions of testing.

Table 5.

Summary of Deformation Structure and Phases Present for Deformed Alloys

Alloy Testing Loading $\sim \%$ $e$ $\alpha'$ Deformation Temp. Strain (hcp) (bcc) Twins 304 297 impact 9 present present none 304 297 slow 9 present present none 304 76 slow 9 present present none 304 76 slow 9 present present none 306 76 slow 3 present present none 310 297 impact 9 none none none 310 297 slow 3 none none none 310 297 slow 3 none none none 310 297 slow 3 none none none	~ Dislocation Density (cm <sup>-2</sup> )	1011	$10^{11}$	$10^{11}$	10 <sup>9</sup>	1011	1 0 <sup>11</sup>	$10^9$	10 <sup>9</sup>
Alloy Testing Loading $\sim \%$ e $\alpha'$ . Temp. Strain (hcp) (bcc) 304 297 impact 9 present present 304 297 slow 9 present present 304 76 slow 3 present present 304 76 slow 9 present present 304 76 slow 3 present present 310 297 impact 9 none none 310 297 slow 3 none none 310 297 slow 3 none none	Deformation Twins	present	none	none	none	none	none	none	none
AlloyTestingLoading $\sim \eta_0$ $\varepsilon$ Temp.Temp.Strain(hcp)304 $297$ impact9present304 $297$ slow9present30476slow9present30476slow9present304297slow9present310297impact9none310297impact3none310297slow3none310297slow3none	a' (bcc)	present	present	present	present	none	none	none	none
AlloyTestingLoading $\sim \%$ Temp.Temp.Strain304 $297$ impact9304 $297$ slow930476slow330476slow3310 $297$ impact9310 $297$ slow3310 $297$ slow3310 $297$ slow3310 $297$ slow3	e (hcp)	present	present	present	present	none	none	none	none
Alloy Testing Loading Temp. 304 297 impact 304 297 slow 304 76 slow 304 76 slow 310 297 impact 310 297 slow 310 297 slow	$\sim rac{70}{5}$ train	6	6	6	ŝ	6	6	3	ŝ
Alloy Testing Temp. 304 297 304 297 304 76 304 76 310 297 310 297 310 297 310 297	Loadıng	impact	slow	slow	slow	impact	slow	impact	slow
Alloy 304 304 304 304 310 310 310	Temp.	297	297	76	76	297	297	297	297
	Alloy	304	304	304	304	310	310	310	310

#### 6. Discussion

6.1 Impact-Fatigue Equipment

A new apparatus, designed to simulate the application of repeated variable loads at high strain rates on controlled specimens, has been employed in this study. The test results, using this "impact-fatigue" machine, have proven very consistent. Applied stress versus number of cycles to failure (S-N) curves blend very well with conventional "push-pull" or flexure fatigue curves (Figure 15). Since only ductile specimens(AISI 310, 304) were tested, it is of considerable interest to employ this equipment in the study of the characteristics of higher strength aerospace alloys. Presumably, crack propagation behavior under impact loading would vary considerably from high cycle fatigue loading conditions.

#### 6.2 Fatigue Strength

Apparently, no previous fatigue studies of austenitic stainless steels have included AISI 310; this investigation represents the first documentation of AISI 310 in the temperature range 76 to 297K. As Figures 4-6 suggest, the fatigue strength increases with decreasing test temperature and is stronger for uniaxially-applied impact loads. While the ratio of the yield strength at 76K to the yield strength at 297K is about 2.6, the ratio of the increase of fatigue strength between the two temperatures is considerably less, only about 1.6. This ratio decreases more if the impact stresses are applied biaxially or triaxially.

Triaxial AISI 310 endurance limits are less than the corresponding yield strengths at each temperature. In fact, all triaxial S-N curves appear to merge at longer fatigue lives and to have endurance strengths of about 20,000 psi at over  $10^4$  cycles. This strength represents the lowest design stress level found for AISI 310 and is independent of

operating temperature in the interval 76 to 297K.

Fatigue strengths of AISI 304, (Figure 7-9) exhibit the same trends as AISI 310. In general, however, at given fatigue life, temperature, and stress conditions, the fatigue strength of AISI 304 is slightly higher. Also, AISI 304 endurance limits are better, with the minimum endurance strength of about 25 to 30,000 psi under triaxial loading conditions at all temperatures. Again, under triaxial conditions at longer fatigue life, the effect of temperature is insignificant.

Compared to tensile yield strengths, the fatigue strength in uniaxial and biaxial conditions increases more as the test temperature is lowered (yield strength ratio, 76 to 297K, of about 1.3; fatigue strength ratio, 76 to 297K, of about 1.7). This is opposite to the effect of temperature on AISI 310 yield strength and fatigue strength ratios. But, the ratio of fatigue strength increase with decreasing temperature for the two alloys is nearly identical (AISI  $310 \approx 1.6$ , AISI  $304 \approx 1.7$ ). This implies that the effect of martensite formation on fatigue life is relatively insignificant; in AISI 304 significantly more martensite forms in the 76K specimens than forms in the 297K. This conclusion is startling since purer martensitic alloys (Fe-Ni alloys, AISI 400 series) are well known to exhibit more brittle behavior at lower temperatures.

Even under triaxial loading conditions, when the effect of temperature becomes insignificant, (implying some embrittlement at lower temperatures) the argument cannot be made that martensitic transformation is responsible; the same trend is found in AISI 310, where no martensite was observed (see Table V)! As AISI 310 yield strengths are equivalent or better than AISI 304 tensile yield strengths, the slightly better fatigue strengths obtained in AISI 304 may be attributed to martensite formation. The same explanation is usually

presented for the higher ultimate strengths obtained in AISI 304, compared to AISI 310 at 76K (see Table IV). Therefore, after examination of AISI 310 (which has no stress-induced transformation) and AISI 304 (which has a stress-induced transformation) impact-fatigue data, no deleterious behavior can be attributed to the martensitic transformation. Reduction of endurance strengths under triaxial loading conditions is almost identical in both alloys, indicating that face-centered cubic alloy dislocation interactions or deformation twins are responsible, not martensitic transformations.

### 6.3 Welding Effects

In the course of this program,other variables such as weld properties, the effects of cold work, and the effects of thermal cycling between 76 and 297K (to produce about 5% martensite in AISI 304) were examined. Some of the results are briefly discussed below.

If AISI 304 is carefully welded, using the heliarc process, impact-fatigue strengths of the weld area are approximately 15% lower than the parent material strengths (see Figures 10 and 11). Weld tensile strengths (Table IV) are also lower than parent material strengths, as fractures occurred in the weld portion of specimens. Fatigue strengths at  $10^4$  cycles to failure are compared in Figure 14 for AISI 304 (welded), AISI 304 (annealed) and AISI 310 (annealed). Under biaxial loading conditions, the welded AISI 304 is about 15% weaker than the annealed materials. Unfortunately, it was not possible to manufacture triaxial specimens (round, notched) from the 1/8 inch thick sheet specimens. But the data of Figure 14 do imply that the weakest portion of a welded, AISI 304 structure would be that section which is welded and exposed to triaxially oriented loads under impact conditions. At  $10^4$  cycles, the impact-fatigue strength of such sections would be about 20,000 psi or greater. The endurance limit of welded,

triaxially loaded specimens, however, would likely be less, 10,000 psi being a reasonable, though conservative estimate. One can arrive at this minimum stress estimate by extrapolating the welded uniaxial and biaxial fatigue strengths to triaxial strengths, using the trend of the annealed AISI 304 fatigue strengths as a guideline.

## 6.4 Thermal Cycling Effects

It was thought that thermal cycling to induce martensite formation spontaneously would, perhaps, alter the impact-fatigue behavior. Earlier, we had shown that prior transformation in polycrystalline AISI 304 reduces the "easy glide" portion of the stress-strain curve at 76K. <sup>5</sup> This "easy glide" region was shown to correspond to formation of hexagonal close packed martensite during plastic straining. Perusal of Figures 7 and 8 indicate that previous formation of small amounts of martensite has no detectable effect on subsequent impactfatigue life.

#### 6.5 Cold Working Effects

Cold working of AISI 310 or 304 at 76K significantly increases the subsequent impact-fatigue properties at 76 and 297K (see Figure 17). It is interesting to compare the ratios of the fatigue strengths (at 10<sup>3</sup> cycles) of the cold worked to annealed conditions. Both AISI 304 and 310 at 76 and 297K have ratios (fatigue strength, cold worked/ fatigue strength, annealed) of between 1.4 and 1.5. This again strongly implies that martensite formation has little effect on fatigue behavior, since cold working of AISI 310 produced no martensite, while cold working of AISI 304 produces about 12% hexagonal close packed martensite and 50% body-centered cubic martensite (see Reed, Guntner for martensitic product dependence on applied stress or plastic strain).

The data contained in Figures 15 and 16 indicate the high degree of correlation between our results and the conventional fatigue test results of others. <sup>15-22</sup> It proves informative to compare Figure 15 with the tensile results of Figure 40. At one cycle, equivalent to a high strain rate tensile test, the "fatigue" strength corresponds very well to the tensile strength. At lower applied stresses (higher cycles to failure) the endurance strength falls to approximately the minimum yield strength (20,000 psi).

#### 6.6 Fatigue Fracture Stress

If the data is converted to fatigue fracture stress (applied load/ reduced area at fracture), the temperature dependence of the data is considerably less. In particular the biaxial and triaxial results for both AISI 304 and 310 are essentially independent of temperature. Slight temperature dependence is shown by the uniaxial data.

#### 6.7 Deformation Energy Density

Consideration of the energy density curves in Figures 26-33 leads to some interesting speculation. It is clear that as the impact load is decreased, providing longer fatigue life, the energy absorbed by the specimen prior to fracture is reduced toward zero. For this to occur, localized deformation must replace more uniform, "tensiletype" deformation. That is, it is very likely that crack initiation processes are changing as a function of applied load. Although for simplicity the energy density-cycles to failure curves are characterized as a linear function in Figures 26-33, it is very probable that the function becomes asymptotic to zero load as the number of cycles to failure continue to increase. Following this, it is reasonable to speculate that zero energy density represents the material endurance limit; impact loading, below the stress necessary to produce some finite specimen strain, will never produce specimen fracture. Of

further interest is the tendency of all assumed linear functions (energy density versus cycles to failure), if extrapolated, to converge to zero energy density at the same number of cycles to failure (see Figures 27, 28 and 31, for example).

6.8 Martensite Formation in AISI 304

Martensite formation was monitored as a function of number of cycles, and the results are presented in Figure 36. The increase of martensite (a ferromagnetic phase) with increasing number of fatigue cycles promotes the feasibility of monitoring fatigue life. The undeformed material, austenite, is paramagnetic; therefore, magnetometric monitoring of specimens or components exposed to repeated loads should very easily indicate impending fracture.

The monitored specimen, tested at room temperature, exhibits an unusual increase in uniform reduction in area, elongation, and percent martensite transformed just prior to fracture (Figure 36). To our knowledge, the rather sudden increase in uniform specimen strain (not specimen necking) has not been reported in the literature and is quite unexpected. Further consideration and documentation of this is needed. Arguments cannot be advanced that fracture propagation is solely responsible, since little uniform martensite transformation would be expected to occur except along fracture paths.

6.9 Tensile Data

Compiled tensile data for both alloys, shown in Figure 39 and 40, indicate the increase of AISI 310 yield strength, compared to AISI 304, at lower temperatures. Again, the lower 304 yield strength can be attributed to hexagonal martensite formation. Higher 304 tensile strengths have been attributed to body-centered martensite strengthening. Further discussions of these contributions are contained 5, 29, 30
Figure 41 contains data on martensite (b.c.c.) formation as a function of specimen strain for comparison of impact-loaded and tensile-loaded specimens. Room temperature data of Alloys A and B do show significant difference. Both of these alloys are commercially classified and sold as AISI 304, but it is easy to discern their different transformation characteristics. This difference is also apparent at 76K. Comparison of the effect of strain rate on transformation tendency is not as conclusive. From our available data, no definitive statement can be made related to this question, although many others claim that increased strain rate produces lower amounts of martensite 3, 9, 24, 25

# 6.10 Microscopy

Discussion of optical and electron microscopy results is presented in the Experimental Results section. Several observations provide points for additional discussion. All specimens examined had necked prior to crack propagation and fracture. In this sense, therefore, fracture was very similiar to conventional tensile fracture, as contrasted to fatigue crack propagation and fracture. Martensite played no detectable role in crack propagation, nor were premature cracks initiated by the martensitic structures.

It is well documented that high energy rate forming of facecentered cubic metals produces an abundance of deformation at ambient temperatures or higher. Similarly, impact-loading of AISI 304 produced deformation twins (see Table V), however, none were 31 found in 310. As suggested by many (see Venables for review), this evidence supports the view that lower stacking fault energy promotes twinning. At lower temperatures in 304, martensitic transformations (hcp and bcc) evidently replace twinning as deformation products.

# 6.11 Possible Additional Programs

There are indications <sup>(32)</sup> that the fatigue strengths of AISI 300 Series steels which undergo partial martensitic transformations are less at 20K in the large amplitude range than at 76K. Therefore, it should be clearly understood that the conclusions drawn in the report only apply in the temperatures range 76 to 294K. Additional data should be acquired below 76K to enable accurate prediction of the long-time life of components exposed to periodic sudden loads in this temperature regime.

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# Appendix

The following paper, "Apparatus for Impact-Fatigue Testing," describes the modifications made to automate a standard impact machine. It has been accepted for publication in Section C, Journal of Research, NBS (1971).

Another paper summarizing the main findings in this report is presently in progress. Apparatus for Impact-Fatigue Testing

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A standard impact machine was extensively modified to allow the measurement of the response of specimens to repeated, controlled impact pulses. This equipment enables one to vary the temperature (76 - 297K), specimen geometry (uniaxial, biaxial, triaxial stress systems), and load levels. At stress levels in the neighborhood of the yield stress on the order of 10,000 impact cycles are needed to fatigue specimens to fracture. Strain rates achieved are moderately high, of the order of 1000 min<sup>-1</sup>, which conveniently form intermediate data between tensile (max. of about 100 min<sup>-1</sup>) and explosive straining data (about 6000 min<sup>-1</sup>). Contrasted to standard fatigue tests, no constraint is placed on specimen elongation and only unidirectional stresses are imposed. Typical impact-fatigue results for AISI 310 stainless steel are presented.

Key words: Cryostat; fatigue; impact; low temperature; mechanical property equipment; stainless steel.

<sup>\*</sup> This work was carried out at the National Bureau of Standards for the Naval Air Engineering Center, Philadelphia, Pennsylvania.

## 1. INTRODUCTION

In many low temperature applications such as in the transportation of dewars or in the transfer of cryogenic liquids through pipe, system components experience periodic sudden jolts or loads. Prediction of expected life of these components can best be accomplished by testing controlled specimens under repeated impact loads which approach up to the yield load. Such a test, then, can be described as impact-fatigue, as the specimen eventually fractures in a manner similar to conventional fatigue fractures.

To perform such tests a standard commercial impact machine was extensively modified. These modifications resulted in an apparatus that could repeatedly apply sudden loads in the range 0 to 1800 kilograms for the many repetitions sometimes required for specimen fracture. Additionally, the specimen chamber was insulated to allow temperature environments from 76 to 297K. Our paper describes this new equipment.

A standard Riehle model PI-2<sup>\*</sup> impact testing machine was available. This machine has three hammer sizes, 15, 30, and 60 lbs. (6.8, 13.6 and 27.3 kg) and the hammer drop is continuously adjustable from 0 to 48 inches (0 to 1.3m). Our impact-fatigue life determinations, however, would require greater than 20,000 cycles in some cases so it became obvious that the single cycle operation for which this machine was designed was not feasible. We designed major modifications to automate the repetition, to allow tests to be conducted at cryogenic temperatures, and to permit the use of specimens in a great variety of configurations. Stress pulses are applied to the specimen in one direction, while the conventional fatigue tests are typically either "push-pull" with alternating tensile and compressive forces or flexure with alternating applied bending forces. Another feature of the impact-fatigue test is that the specimen is not constrained with respect to specimen strain.

\* The use of trade names in this paper in no way implies endorsement or approval by NBS and is included only to define the experimental procedure.

#### 2. MODIFICATIONS

Figure 1 a is a schematic view of the impact-fatigue tester. A 1/6 hp, 1725 rpm electric motor is geared to rotate a 24 inch diameter wheel at 7.3 rpm. The shaft and bearings which support this wheel are clamped to the upright part of the impact machine frame by U-bolts. A brass hammer release arm protruding through the outer edge of this moving wheel serves to pick up the hammer and raise it to a predetermined height. At this point, the trip arm (Fig. 2) encounters a block which is supported behind the rotating wheel. The trip arm rotation causes a cam motion which swings a locking bar out of position allowing the hammer release to simply fall back under the weight of the hammer. A second trip block is at the top of the wheel's revolution to insure that the system does not bind there as it passes the pendulum shaft. After each trip, springs pull the release bar back into a locked position to repeat the cycle.

The standard anvil on the base of the impact machine has been closed off on the right end to serve as the fixed point to which the right specimen pull rod is anchored, either directly or through a load cell (Fig. 3). The left pull rod passes through a three piece impact yoke assembly held together by eight  $\frac{1}{4}$  inch stainless steel bolts. The hammer impact is absorbed by this yoke which transmits it to the specimen, since it is free to move in a right-left direction on four rollers. When the sample breaks, the yoke moves to the left and allows the hammer to strike a switch on the base which removes electrical power from the whole system.

A solenoid system prevents secondary impact by the recoiling hammer (Fig. 1). This stop is a 3/4 inch round bar which rides in a brass bushing inserted into a drilled hole in the impact machine. When this bar is in the fully inserted position, the impact hammer cannot swing past it to strike the yoke. A portion of the bar has a flat depression ground into it so that when the solenoid is activated and the bar partially withdrawn from the anvil the hammer may pass by freely. A switch on the stationary wheel near the block that trips the hammer release mechanism controls the power to the solenoid. As the





fatigue tester.



Figure 2. Impact hammer release mechanism. The trip arm is rotated by a stationary block fixed at the desired position behind the 24 inch wheel. This allows the hammer release to fall back under the hammer weight. Springs then return the mechanism to a locked position.



Figure 3. Specimen impact system.

trip table (Fig. 2) passes by this switch, the secondary stop is removed from the impact hammer path. By the time the hammer has recoiled, power has been removed from the solenoid and springs have pushed the stop back into position. A pin through this bar operates another switch to advance a cycle counter.

Wherever possible, the springs in the hammer-release mechanism were doubled, so if one fails during a test the second will continue to function until repairs can be made. It is important to use high quality steel springs to minimize difficulty due to breaks; soft, tempered springs were found more reliable than high strength units. Since this modification results in a much higher repetition rate than was originally intended, the welds between the pendulum shaft and fittings on both ends were reinforced.

## 3. CRYOSTAT

The cryostat used for 76K tests consisted simply of a 2 inch diameter Teflon rod, 7 inches long, with a 1 inch bore through the center. The specimen pull rods are supported and centered by Teflon end caps screwed into the main chamber (Fig. 3). Rubber O-rings in both of these caps served as an initial seal around the pull rods to minimize the loss of liquid nitrogen. However, in cooling to 76K, the Teflon contracts considerably and then serves as the main seal. Because of this great contraction, it is necessary to check for free movement of the pull rods when they are at test temperatures. Two small holes in the side wall serve for filling with liquid nitrogen and venting. A liquid flow of about  $6 \frac{1}{h}$  was generally used to maintain temperature. At room temperature this cryostat was still used to assure the proper positioning of the sample.

For 195K tests, a slight modification of this system was employed. Instead of the small holes in the side wall for filling and venting nitrogen, a  $1 \times 4$  inch panel was removed. A styrofoam box was attached to act as a reservoir for the powdered dry ice used to fill the cryostat. Consumption in this case was about 0.51b/hr.

#### 4. DISCUSSION

We used a load cell specially constructed to withstand sudden impacts and a high speed recorder to measure impact loading. When using the 30lb. (13.6kg) hammer, the impulse time is about 2 msec. Load cell response (Fig. 4) indicates a damped ringing, but this is a characteristic of the load cell. While the specimen probably does vibrate in the uniaxial direction, our system is incapable of measuring the compressive forces it may experience, but they are believed to be very small.

During the progress of a test the specimen undergoes plastic strain. This allows the yoke to move in the direction of motion of the impact hammer. Thus, by measuring the position of the yoke frequently during a test, it is possible to determine specimen strain as a function of the number of cycles. Naturally, this function depends on variables such as material, temperature, and applied pulse. Figure 5 illustrates a typical curve of strain versus number of cycles for an AISI 310 stainless steel specimen. Initially, the specimen undergoes considerable strain but, after sufficient work hardening (several hundred cycles), its length becomes nearly constant.

Since each cycle corresponds to a 2 ms impulse time, the coordinate giving the number of cycles is also a time coordinate and its slope gives the strain rate. With our experimental conditions, the maximum initial strain rate was 2100 min<sup>-1</sup>. This is conveniently intermediate between the maximum strain rate in tensile tests of about 100 min<sup>-1</sup> and the approximate lower limit<sup>[1]</sup> in explosive straining of 6000 min<sup>-1</sup>.

From the strain readings and load calibration, a typical stress-strain curve may be constructed. Figure 6 shows this curve for AISI 310 at 297K. The stress used to construct this curve is the engineering stress, i.e., load divided by original area. This stress level is constant throughout the test.



Figure 4. Time response curve recorded by load cell and high speed recorder.



Figure 5. Specimen strain during impact-fatigue life.

By varying the height of the hammer (stress amplitude), a typical fatigue (S-N) curve of stress level versus number of cycles to failure may be obtained. The shape of such curves for AISI 310 at 297, 195 and 76 K, as shown in Fig. 7, is very similar to those obtained by conventional fatigue tests in which the high strain rates are not achieved and either the load or specimen deflection amplitude is held constant.

Any specimen configuration, limited only by length (about 5 inches), may be measured. In practice, we have measured both uniaxial (tensile), biaxial, and triaxial (notch tensile) specimens of both AISI 304 and 310 stainless steels at 76, 195 and 297K. These test results will be published later.

## 5. REFERENCES

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Figure 6. Typical stress-strain curve for AISI 310 stainless steel showing repeated impact cycles at 297K (1 kg/mm<sup>2</sup> =  $9.8 \times 10^6$  N/m<sup>2</sup>). Compressive stresses due to recoil of specimen upon itself are very small and not shown.



Figure 7. Fatigue life curve for AISI 310 at 297, 195, and 76 K.

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16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)

Two 300 series stainless steels were tested for impact-fatigue life and tensile properties as a function of temperature, percent transformed phase (martensite), stress level, and specimen geometry. These alloys were studied to predict the fracture characteristics of parent material and weld joints in large cryogenic dewars which are subjected to periodic stresses. Normally, AISI 304 is employed in the construction of such dewars. Under fatigue loading conditions at cryogenic temperatures, the behavior of AISI 304 is complicated by the gradual transformation to a martensitic structure. The influence of this transformation on the fracture characteristics was studied. For this purpose, a new impact-fatigue test and necessary equipment were developed.

The fatigue strength of AISI 304 and 310 exhibit similar trends, but at a given fatigue life and temperature, the fatigue strength of AISI 304 is slightly superior. AISI 310 displays an endurance limit of about 20,000 psi <sup>\*</sup>; the 304 limit is 25-30,000 psi. However, the lowest endurance limit (weakest) specimens appear to be the triaxially-loaded AISI 304 weld specimens which have an endurance limit near 10,000 psi. Little temperature-dependence of the impact-fatigue properties was observed. \* For ease in interpretation all stress values in the text are expressed in units of psi. Conversion to other stress units are

 $10^3 \text{ psi} = 0.703 \text{ kg/mm}^2 = 6.90 \times 10^6 \text{ V/m}^2$ 

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