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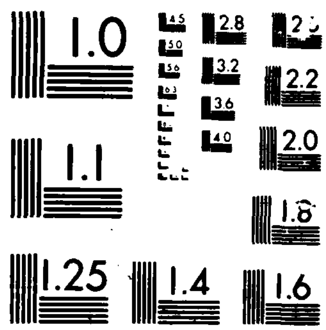
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STUDY OF EFFECTS OF ALLOYING AND HEAT TREATMENT ON HYDROGEN EMBRITTLEMENT SUSCEPTIBILITY OF ELECTROSLAG REMELTED 4340 STEEL

October 1986

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FINAL REPORT

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ABSTRACT

This program focuses on the need to improve the resistance of high-strength steels to hydrogen embrittlement or hydrogen stress cracking. Variations in heat treatment and modifications in alloy composition of electroslag remelted 4340 steel at 53 HRC were extensively explored. Target goals were established in terms of a threshold stress intensity parameter, KIscc for open circuit potential conditions, designated KIhem for cathodic charging conditions under stress during test, which were the primary test conditions in this program.

In general, all alloy additions improved KIhem. The addition of 0.1% vanadium appears to be the most significant individual modification to ESR 4340 steel in that it alone provides the same gains as the more heavily alloyed ESR 4340 steels. The results must be somewhat qualified because the hardness of ESR 4340V was 50 HRC instead of the intended 53 HRC. Silicon additions of about 1.5% tended to maximize the benefits from alloy modifications. Although within an alloy system the heat treatment effects were minor or secondary relative to alloy additions, the use of an intermediate quench and subzero cooling appeared to maximize the benefits from heat treatment. Increasing the threshold KIhem was not directly related to tempering temperature as anticipated. Overall, KIhem was increased from 10 ksi SQR(in.) to a maximum value of about 15 ksi SQR(in.). Other improvements must be based on nonconventional approaches to thermal processing.

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TECHNICAL REPORT

1.0 INTRODUCTION

Ballistically resistant advanced helicopter steel components can use either high-strength or high-hardness (53HRC) ESR-steels, or pay a weight penalty and use thicker, lower-hardness (43HRC) ESR-steels. Reoccurring hydrogen embrittlement problems in fracture critical members is the reason many designers are making this change and paying the weight penalty. Because of the extensive use of high-strength steels in current designs, the solution of going to lower-strength steels has limited viability, until the weight penalty becomes too severe. These weight penalties can only be avoided by improving the hydrogen embrittlement resistance of high-strength or high-hardness (53$\pm$1HRC) ESR 4340 steel.

The susceptibility to hydrogen assisted stress cracking or hydrogen embrittlement is often measured in terms of a threshold stress intensity parameter under conditions of stress corrosion cracking, KIscc. Implied is testing under conditions of an open circuit potential in an aqueous solution. During test, cathodic charging conditions are used that represent conditions of galvanic coupling often found in a service environment. A -1.2V potential is appled that simulates the sacrificial anodes of a zinc coating on steel. Because of this difference, the threshold stress intensity parameter under conditions that produce hydrogen embrittlement is designated as KIhem for the purpose of this report. Under these conditions, atomic hydrogen is produced on the surface while the sample is under stress.

Other authors report the data as KIscc as a function of potential, but enough data exits to support the contention that they should be treated separately. Often KIscc and KIhem are identical, but other times the difference is significant enough to influence alloy selection, as will be illustrated in this report.

The objective of this program is to find an alloy modification or heat treatment that would increase the resistance to hydrogen embrittlement or the hydrogen stress cracking threshold of ESR 4340 steel at 53HRC. Quantitatively, in terms of the threshold stress intensity parameter for hydrogen assisted stress cracking (KIhem), the goals can be identified from the results on a split heat of 4340 steel (Ref 1), where one half the ingot was vacuum arc remelted (VAR) and the other half was electroslag remelted (ESR). Schematically the measured threshold stress intensity results for hydrogen stress cracking, recently presented at a

Sagamore conference (Ref 2) are listed in the following sketch illustrating projected improvements from 10 to 43 ksi.SQR(in) as the hardness is dropped and VAR is used instead of ESR.

THRESHOLD STRESS INTENSITY, KIhem

53HRC				43HRC		
ESR	==>	VAR	==>	ESR	==>	VAR
10	==>	15	==>	29	==>	43

2.0 TECHNICAL APPROACH

It is the intent of this program to use a selected group of commercially available alloy ESR 4340 type steels that have been relatively well characterized to address the use of alloy modifications or heat treatment to improve the resistance to hydrogen assisted stress cracking instead of melting special heats of steel. A matrix of five materials and four heat treatment conditions were used to evaluate the problem.

The five materials represent two basic alloy steel modifications: (1) increased silicon additions, and (2) increased amounts *of carbide formers* to effect the secondary hardening characteristics. Alloying additions were considered because their general effect is to require a higher temperature for a given holding time to secure a given hardness, thus potentially permitting a greater stress relief, when compared to conventional carbon, quenched and tempered steels. The generic guideline being evaluated was that higher tempering temperatures produce increased resistance to stress corrosion cracking for the same hardness level of a tempered martensitic steel.

Modifications in silicon were selected to represent non-carbide forming alloying elements. Increased amounts of silicon are known to improve the resistance of a quenched steel to softening. Amounts varying from as much as 0.5% to 2.0% have been shown to increase the hardness after tempering by as much as 4HRC. Therefore, three of the five steels selected represents one grouping designed to illustrate the effect of increased amounts of silicon from 0.25%, to 1.5% to 2.5%. Conventional alloys steels of ESR 4340 (baseline), ESR 4340M or ESR 300M, and the new ESR HP310 alloy steels, respectively, were used to represent this variation in silicon content.

The presence of appreciable amounts of strong carbide forming elements, such as chromium, molybdenum or vanadium may cause softening to be retarded, or may result in an actual increase in hardness when tempered over a certain range of temperatures. This "secondary hardening" effect should provide a greater hardness for a given tempering temperature than would be obtained with a lower alloyed steel (such as 4340 steel). Therefore, a second grouping reflects an increase in the amount of carbide formers from the conventional ESR 4340 to ESR 4340V that has an additional amount of 0.1% vanadium. The third ESR steel in this group is commercially designated as D6Ac steel. In addition to the 0.1% vanadium, D6Ac also has an increase in the carbide formers of chromium and molybdenum, with a concurrent decrease in the amount of nickel. The increase in molybdenum is almost negated by the decrease in nickel, when the effective resistance to softening of these alloying elements is compared.

Four treat conditions were evaluated to address the effect of altering either the residual stress or the amount of retained austenite, which might in turn further effect the resistance to hydrogen assisted stress cracking. In addition to the normal quenched and tempered heat treatment, marquenching was used as an alternative design to reduce the amount of residual stress. Since this delayed quenching treatment might also increase the amount of retained austenite, a liquid nitrogen quench was used as a subzero cooling treatment to complete the austenite to martensite transformation. This treatment was applied as an alternative to both the conventional and marquench heat treatments. Retained austenite as a factor that inhibits or accelerates hydrogen assisted stress cracking has not been clearly established.

Abnormal quantities of retained austenite are affected by the combined action of increasing alloying content, and an excessively high quenching temperature. It should also be recalled that coarse grain and the presence of austenite stabilizing elements, such as nickel and manganese, also favors the retention of austenite. Transformation of retained austenite upon tempering at a given temperature will be in accordance with the isothermal transformation at that temperature level. Treatments to reduce residual stresses tend to increase the amount of retained austenite.

This program addresses the factors affecting susceptibility to hydrogen stress cracking not from a completely research mechanistic analysis, but from a screening survey type program to identify significant processing parameters.

3.0 EXPERIMENTAL PROCEDURE

3.1 Equipment: Instead of the conventional test method to determine the threshold KIhem, a modified, low-cost technique was employed. The proposed method uses the rapid, inexpensive, modular (RIM) SCC-testing system. Only a maximum of five Charpy-Sized specimens are required to obtain measurement of KIscc instead of 13 or more large size cantilever beam or wedge opening load (WOL) specimens, conventionally used to obtain one measurement of threshold KIscc or KIhem, as per our designation.

The time of test is also 8-hrs for the RIM SCC-testing system as compared to as many as 5,000hrs per run-out on a conventional cantilever beam or WOL test.

3.2 Specimens: Charpy-sized specimens will be used in all cases. The RIM SCC-test method is described in Ref.3. The paper provides background detail of the test method utilized to minimize program cost. A machined (crush ground) notched surface was used instead of a fatigue precracked notch. The depth of the notch was 2mm (0.078in). The root radius was about 0.1mm (3-4mils). A 300F stress relief was employed immediately after all grinding operations.

The test specimen orientation was LT in all cases. As per ASTM E399, the specimen orientation is designated by two letters with the first letter (L) designating the direction of the normal to the crack plane and the second letter (T), designating the crack direction, where L, T and S (short transverse) are the orthogonal directions of the ingot.

3.3 Test Method: To measure KIhem with the RIM SCC-testing system, one specimen is loaded to fracture to obtain the maximum fracture load. If the fracture load is less than the ultimate tensile strength of the material in magnitude; i.e., 250lb for a 250ksi steel, then the specimen is fracture toughness critical. The full scale load on the chart is then adjusted to be slightly more than the fracture load.

A second specimen is then placed in a hydrogen producing environment, which is a 3.5% salt water solution maintained at -1.2 volts vs a saturated calomel electrode. Conventionally, the specimen is then step-loaded until crack initiation occurs. Initial step-loads of 50, 65, 75, 80, 85, 90, 95, and 100% of the fracture load are used. The specimen is held for one hour at each step. A third specimen is then used to refine the value of the crack initiation load, P_i; for example, if the second specimen initially cracked at 75% of the fracture load, then to more accurately measure the crack initiation load, step-loads of 50, 65, 68, 70, 72, 74, 76 and 78% are used. The threshold KIhem is then estimated as three tenths of the initiation load, Pi.

Since five specimens did not exist for each test condition, the applied step-loads were modified for this program. A large number of small step increments were used with the limited number of samples available; the fewer the samples, the smaller the increments, the larger the number of steps, the longer the duration of the test, which is equivalent to decreasing the strain rate in a slow strain rate tensile test.

The fracture toughness per ASTM E399 can be measured either by conventional slow strain rate techniques to measure KIc or dynamically with an instrumented impact test machine to measure the dynamic fracture toughness, KId. Once crack initiation occurs or Pi is measured, the crack can be extended by fatigue to a crack depth ratio of one-half. To insure the specimen were free of hydrogen, the test specimens were baked prior to fatigue precracking.

3.4 Organizational Flow Chart: The flow chart shown in FIG. 1 details the steps of the test program. Each of the five alloy ESR steels had ten Charpy specimen blanks for a total of 50 specimens.

Five specimens from each alloy steel had a standard or conventional quench after austenitizing. Three of these specimens had a conventional temper to 52-54HRC. The remaining two specimens had an additional liquid nitrogen quench and a subsequent temper to 52-54HRC.

Five specimens from each alloy steel were marquenched after austenitizing. Three of these specimens had a conventional temper to 52-54HRC. The remaining two specimens had an additional liquid nitrogen quench and a subsequent temper to 52-54HRC.

The total of 50 Charpy size specimens had the modified, crush ground notches machined to a radius of 3-4mils. These specimens were all stress relieved at 300F immediately after machining.

The total of 50 Charpy size specimens were tested in the RIM SCC-testing system by step-loading in a 3.5% salt water solution at a potential of -1.2V vs a saturated calomel electrode.

After KIhem was measured from the initiation load, each of the specimens were heated to 300F for 8-hrs to remove an residual hydrogen. The specimens were fatigue precracked to such that the ratio of the fatigue length to the specimen depth equals one-half.

From each lot of five specimens, three standard oil quenched and tempered specimens, two of the three specimens were used to measure KIc and the remaining specimen was used to measure KId. From the remaining two specimens that were marquenched, one specimen was used to measure KIc and the remaining specimen was used to measure KId.

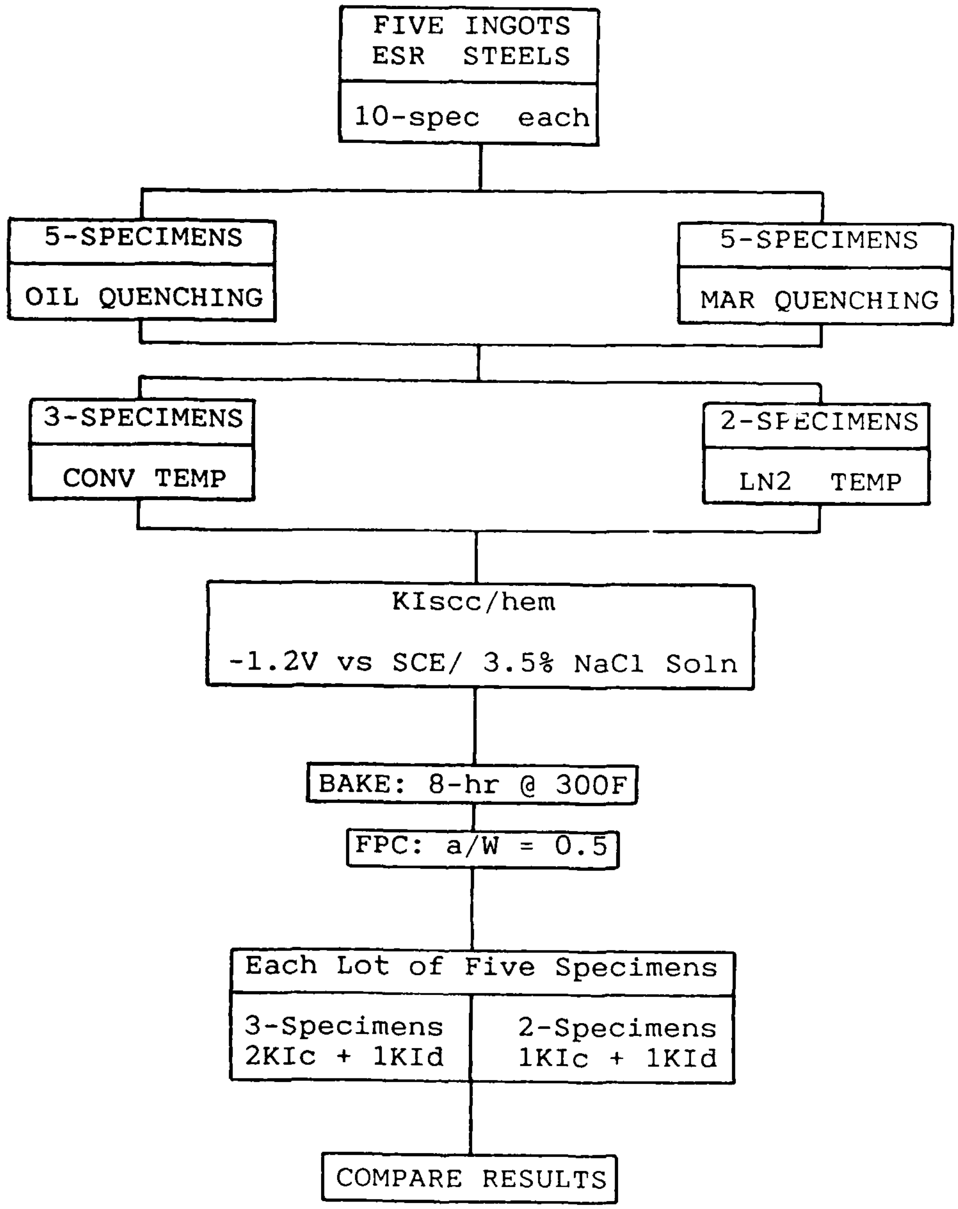


FIG. 1. Flow Chart identifying program elements.

3.5 Sample Identification Chart: The chart shown in Table 1 identifies specific test samples that were used to conduct the tests described in the previous section.

Table. 1.

Specific sample identification chart for each test program element.

AUSTENITIZE/ OIL QUENCH					AUSTENITIZE/ MARQUENCH				
LN2 QUENCH & TEMPER		STD QUENCH & TEMPER			LN2 QUENCH & TEMPER		STD QUENCH & TEMPER		
KIc	KId	KIc	KIc	KId	KIc	KId	KIc	KIc	KId
A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
B1	B2	B3	B4	B5	B6	B7	B8	B9	B10
C1	C2	C3	C4	C5	C6	C7	C8	C9	C10
D1	D2	D3	D4	D5	D6	D7	D8	D9	D10
E1	E2	E3	E4	E5	E6	E7	E8	E9	E10

MATERIAL IDENTIFICATION

S/N A : ESR 4340
S/N B : ESR 4340V
S/N C : ESR 4340M
S/N D : ESR HP310
S/N E : ESR D6Ac

4.0 RESULTS

4.1 Chemical Analyses: The chemical composition of the five alloys as determined spectrochemically, including Leco vacuum fusion analysis for carbon is summarized in Table 2 as S/N A (ESR 4340), S/N B (ESR 4340V), S/N C (ESR 4340M), S/N D (ESR HP310), and S/N E (ESR D6Ac).

For comparison, the composition supplied on the certifications from the steel company or the composition of the experimental heats supplied by the Ingersoll-Rand Company (Ref 4.) are included in the table. Also, any available chemistries from the published literature on VAR have been added. Finally, the AMS specification limits and/or the corresponding military specifications are also identified when available.

Table 2

Chemical composition for five ESR 4340 type alloy Steels

#	STEEL	SOURCE	Fe	Si	C	Mn
1	4340	S/N A (HT #3710046)	BASE	0.27	0.42	0.71
2	4340	HT #3710046 (TR 82-49)	BASE	0.26	0.41	0.70
3	4340	AMS 6415E	BASE	0.20/0.35	0.38/0.43	0.65/0.85
4	4340V	S/N B (HT #N264-IR-1)	BASE	0.24	0.39	0.80
5	4340V	AVG. 13 PROD. HEATS	BASE	0.25	0.41	0.80
6	4340V	MIL-S-8844C CLASS 1	BASE	0.20/0.35	0.38/0.43	0.65/0.90
7	4340M	S/N C (HT #N848-4R-1)	BASE	1.56	0.40	0.88
8	4340M	ESR Exp FIRST HEAT	BASE	1.58	0.41	0.82
9	4340M	ESR Exp 2ND HEAT	BASE	1.51	0.40	0.88
10	4340M	VAR HT #3831573	BASE	1.59	0.43	0.70
11	4340M	VAR HT #3812628	BASE	1.70	0.40	0.73
12	4340M	MIL-S-8844C CLASS 3	BASE	1.45/1.80	0.40/0.45	0.65/0.90
13	300M	AMS 6419	BASE	1.45/1.80	0.41/0.46	0.60/0.90
14	HP310	S/N D (HT #3710234)	BASE	2.50	0.43	1.22
15	HP310	ESR REPUBLIC #3710234	BASE	2.45	0.43	1.04
16	HP310	VAR HT #3811931	BASE	2.46	0.42	0.37
17	HP310	SPEC (NOMINAL COMP)	BASE	2.40	0.40	0.40
18	D6ac	S/N E (HT #791-2R-3-1)	BASE	0.26	0.45	0.85
19	D6ac	ESR Exp FIRST HEAT	BASE	0.24	0.45	0.79
20	D6ac	AMS 6431	BASE	0.15/0.30	0.45/0.50	0.60/0.90

(Table 2 Continued)

#	STEEL	P	S	Al	Ni	Cr	Mo	V
1	4340	0.015	0.002	0.015	1.85	1.00	0.20	---
2	4340	0.008	0.001	0.035	1.73	0.90	0.22	---
3	4340	<0.040	<0.040	---	1.65/2.0	0.70/0.90	0.20/0.30	---
4	4340V	0.004	0.007	0.006	1.93	0.81	0.22	0.07
5	4340V	0.008	0.006	---	1.90	0.85	0.25	0.10
6	4340V	<0.010	<0.010	---	1.65/2.0	0.70/0.90	0.20/0.30	---
7	4340M	0.003	0.002	0.010	1.96	0.87	0.38	0.09
8	4340M	0.004	0.001	---	1.77	0.82	0.37	0.08
9	4340M	0.006	0.002	---	1.84	0.87	0.38	0.08
10	4340M	0.010	0.005	0.094	1.72	0.87	0.40	0.08
11	4340M	<0.008	0.003	0.094	1.80	0.77	0.42	0.09
12	4340M	<0.010	<0.010	---	1.65/2.00	0.70/0.95	0.35/0.45	0.05
13	300M	<0.010	<0.010	---	1.65/2.00	0.70/0.95	0.30/0.50	0.05/0.10
14	HP310	0.013	0.005	0.11	2.20	0.91	0.51	0.27
15	HP310	0.010	0.001	---	1.73	0.86	0.43	0.20
16	HP310	<0.008	0.003	0.070	1.76	0.97	0.36	0.22
17	HP310	<0.010	<0.010	---	1.80	0.90	0.35	0.22
18	D6ac	0.016	0.003	0.009	0.78	1.27	1.05	0.12
19	D6ac	0.008	0.003	---	0.56	1.20	0.98	0.11
20	D6ac	0.010	0.010	---	0.40/0.70	0.90/1.20	0.90/1.10	0.08/0.15

4.2 Hardness after Heat Treatment: The resulting hardness from the prescribed heat treatments are graphically shown in FIG. 2. As noted, the test specimens designated S/N A (4340), S/N C (4340M), and S/N D (HP310), or the grouping representing the silicon additions, all fell within the desired hardness range. It should be noted that HP310 was overtempered (575F recommended) in order to lower the hardness to the range desired for this program. The increased tempering temperature was expected to increase the threshold KIhem.

The test specimens designated as S/N B (4340V) and S/N E (D6Ac), resulted in hardnesses lower than desired. Since the hardness could not be increased without completely repeating the heat treatment, the specimens were tested at the lower hardness levels. The processing plan for each group of specimens is shown in the flow diagrams included in the Appendix. The actual processing schedule is shown in Table 3 with the test results. To be noted is the higher normalizing temperature of 1750F for 4340V(1650F), 4340M (1700F), and D6Ac (1700F). Specified temperatures are in parentheses.

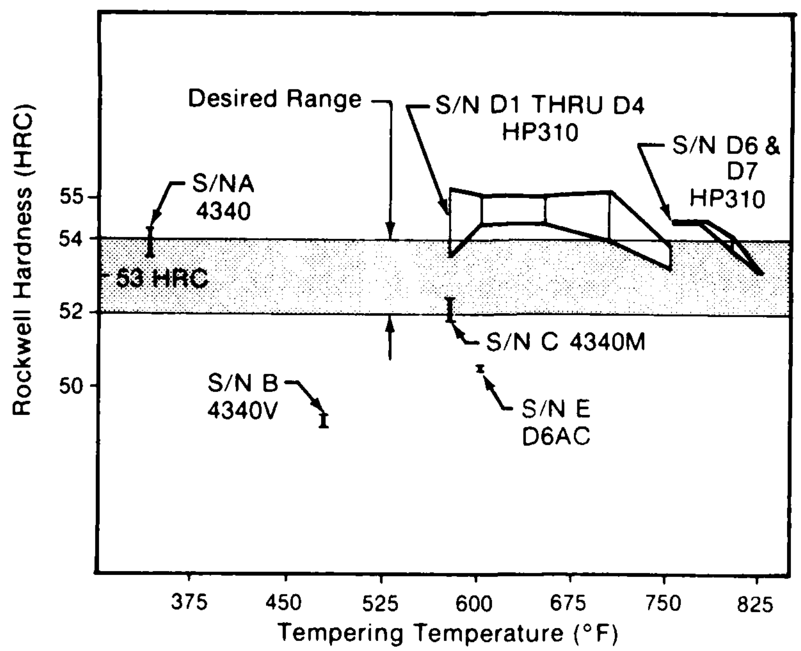


FIG. 2. Resulting hardness from four heat treatments.

4.3 Fracture Toughness and Stress Corrosion Test Results:

Table 3

TEST RESULTS S/N A: ESR 4340

S/N	PC	HRC	UTS	KIhem	KIscc	KIc	KId
A1	NA/ -/ O/ L/ TS	53.7	289	09.7	--	44.8	--
A2	NA/ -/ O/ L/ TS	53.8	290	11.0	--	--	43.4*
A3	NA/ -/ O/ -/ TS	53.2	284	11.1	--	45.8	--
A4	NA/ -/ O/ -/ TS	53.7	289	10.7	--	44.2	--
A5	NA/ -/ O/ -/ TS	53.5	287	10.3	--	--	46.5*
A6	NA/ M/ O/ L/ TS	53.8	290	12.4	--	43.7	--
A7	NA/ M/ O/ L/ TS	54.6	298	11.6	--	--	42.9*
A8	NA/ M/ O/ -/ TS	53.3	285	11.4	--	48.4	--
A9	NA/ M/ O/ -/ TS	53.7	289	10.8	--	47.7	--
A10	NA/ M/ O/ -/ TS	54.1	293	10.6	--	--	41.6*

L E G E N D

S/N = Sample Number

PC = Processing Code

- NA = Normalized @ 1650F (1 hour), and Austenitized @ 1550F (0.5 hour).
- M = Marquenched @ 1000F (0.5 hour).
- O = Oil Quenched to Room Temperature
- L = Liquid Nitrogen Quenched
- TS = Initial Temper @ 340F (2 + 2 hours), and Stress Relieved @ 290F (1 hour).

HRC = Hardness, Rockwell "C" Scale

UTS = Ultimate Tensile Strength (ksi)

KIhem = Hydrogen embrittlement or hydrogen assisted stress cracking, Stress Intensity threshold; ksi.SQR(in). Testing @ -1.2V vs SCE in 3.5% NaCl Solution.

KIscc = Stress corrosion cracking, open circuit Stress Intensity threshold; ksi.SQR(in). Testing @ -0.6V vs SCE in 3.5% NaCl Solution.

KIc = Fracture Toughness per ASTM E399; ksi.SQR(in).

KId = Dynamic Fracture Toughness; ksi.SQR(in).

* = Calculation based on estimate of load.

Table 4

TEST RESULTS S/N B: ESR 4340V mod

S/N	PC	HRC	UTS	KIhem	KIscc	KIc	KId
B1	NA/ -/ O/ L/ TS	49.9	254	13.3	--	63.6	--
B2	NA/ -/ O/ L/ TS	51.5	268	12.5	--	--	45.5
B3	NA/ -/ O/ -/ TS	49.4	250	15.8	--	**	--
B4	NA/ -/ O/ -/ TS	49.0	246	--	37.7	71.1	--
B5	NA/ -/ O/ -/ TS	51.4	267	13.5	--	--	48.4
B6	NA/ M/ O/ L/ TS	49.7	252	18.0	--	69.8	--
B7	NA/ M/ O/ L/ TS	51.8	271	14.4	--	--	50.4
B8	NA/ M/ O/ -/ TS	49.3	249	15.5	--	72.1	--
B9	NA/ M/ O/ -/ TS	51.6	269	12.5	--	84.4	--
B10	NA/ M/ O/ -/ TS	51.5	268	--	**	--	48.8

L E G E N D

S/N = Sample Number

PC = Processing Code

- NA = Normalized @ 1750F (1 hour), and Austenitized @ 1500F (0.5 hour).
- M = Marquenched @ 1000F (0.5 hour).
- O = Oil Quenched to Room Temperature
- L = Liquid Nitrogen Quenched
- TS = Initial Temper @ 475F (2 + 2 hours), and Stress Relieved @ 425F (1 hour).

HRC = Hardness, Rockwell "C" Scale

UTS = Ultimate Tensile Strength (ksi)

KIhem = Hydrogen embrittlement or hydrogen assisted stress cracking, Stress Intensity threshold; ksi.SQR(in). Testing @ -1.2V vs SCE in 3.5% NaCl Solution

KIscc = Stress corrosion cracking, open circuit Stress Intensity threshold; ksi.SQR(in). Testing @ -0.6V vs SCE in 3.5% NaCl Solution.

KIc = Fracture Toughness per ASTM E399; ksi.SQR(in).

KId = Dynamic Fracture Toughness; ksi.SQR(in).

** = Data not obtained; invalid test.

Table 5

TEST RESULTS S/N C: ESR 4340Si mod (300M or 4340M)

S/N	PC	HRC	UTS	KIhem	KIscc	KIc	KId
C1	NA/ -/ O/ L/ TS	53.1	283	14.0	--	49.0	--
C2	NA/ -/ O/ L/ TS	55.0	302	15.2	--	--	31.4
C3	NA/ -/ O/ -/ TS	52.2	275	12.8	--	48.5	--
C4	NA/ -/ O/ -/ TS	51.5	268	--	36.5	**	--
C5	NA/ -/ O/ -/ TS	53.4	286	11.9	--	--	38.3
C6	NA/ M/ O/ L/ TS	52.5	278	13.3	--	46.5	--
C7	NA/ M/ O/ L/ TS	54.6	298	15.9	--	--	**
C8	NA/ M/ O/ -/ TS	51.8	271	12.7	--	49.0	--
C9	NA/ M/ O/ -/ TS	54.4	296	15.3	--	48.7	--
C10	NA/ M/ O/ -/ TS	54.2	294	--	31.0	--	**

L E G E N D

S/N = Sample Number

PC = Processing Code

- NA = Normalized @ 1750F (1 hour), and Austenitized @ 1600F (0.5 hour).
- M = Marquenched @ 1000F (0.5 hour).
- O = Oil Quenched to Room Temperature
- L = Liquid Nitrogen Quenched
- TS = Initial Temper @ 575F (2 + 2 hours), and Stress Relieved @ 525F (1 hour).

HRC = Hardness, Rockwell "C" Scale

UTS = Ultimate Tensile Strength (ksi)

KIhem = Hydrogen embrittlement or hydrogen assisted stress cracking, Stress Intensity threshold; ksi.SQR(in). Testing @ -1.2V vs SCE in 3.5% NaCl Solution.

KIscc = Stress corrosion cracking, open circuit Stress Intensity threshold; ksi.SQR(in). Testing @ -0.6V vs SCE in 3.5% NaCl Solution.

KIc = Fracture Toughness per ASTM E399; ksi.SQR(in).

KId = Dynamic Fracture Toughness; ksi.SQR(in).

** = Data not obtained; invalid test.

Table 6

TEST RESULTS S/N D: ESR HP310

S/N	PC	HRC	UTS	KIhem	KIscc	KIc	KId
D1	NA/ -/ O/ L/ T	53.5	287	13.3	--	37.7	--
D2	NA/ -/ O/ L/ T	53.3	285	13.3	--	--	36.9*
D3	NA/ -/ O/ -/ T	53.2	284	13.8	--	46.0	--
D4	NA/ -/ O/ -/ T	53.4	286	12.7	--	44.7	--
D5	NA/ -/ O/ -/ T	53.8	290	12.7	--	--	39.8*
D6	NA/ M/ O/ L/ T1	53.2	284	11.7	--	39.2	--
D7	NA/ M/ O/ L/ T1	53.1	283	12.6	--	--	30.8*
D8	NA/ M/ O/ -/ TS	53.9	291	12.6	--	48.2	--
D9	NA/ M/ O/ -/ TS	53.9	291	12.4	--	48.0	--
D10	NA/ M/ O/ -/ TS	53.9	291	12.5	--	--	39.6*

L E G E N D

S/N = Sample Number

PC = Processing Code

- NA = Normalized @ 1750F (1 hour), and Austenitized @ 1650F (0.5 hour).
- M = Marquenched @ 1000F (0.5 hour).
- O = Oil Quenched to Room Temperature
- L = Liquid Nitrogen Quenched
- T = Initial Temper @ 575F (2 + 2 hours), and Final Temper @ 750F (1 hour).
- T1 = Initial Temper @ 575F (2 + 2 hours), and Final Temper @ 825F (1 hour).
- TS = Initial Temper @ 575F (2 + 2 hours), and Stress Relieved @ 525F (1 hour).

HRC = Hardness, Rockwell "C" Scale

UTS = Ultimate Tensile Strength (ksi)

KIhem = Hydrogen embrittlement or hydrogen assisted stress cracking, Stress Intensity threshold; ksi.SQR(in). Testing @ -1.2V vs SCE in 3.5% NaCl Solution.

KIscc = Stress corrosion cracking, open circuit Stress Intensity threshold; ksi.SQR(in). Testing @ -0.6V vs SCE in 3.5% NaCl Solution.

KIc = Fracture Toughness per ASTM E399; ksi.SQR(in).

KId = Dynamic Fracture Toughness; ksi.SQR(in).

* = Calculation based on estimate of load.

Table 7

TEST RESULTS S/N E: ESR D6Ac

S/N	PC	HRC	UTS	KIhem	KIscc	KIc	KId
E1	NA/ -/ O/ L/ TS	50.2	257	14.0	--	53.5	--
E2	NA/ -/ O/ L/ TS	53.0	282	10.7	--	--	36.4
E3	NA/ -/ O/ -/ TS	50.5	259	12.3	--	53.5	--
E4	NA/ -/ O/ -/ TS	49.6	252	--	12.4	55.2	--
E5	NA/ -/ O/ -/ TS	52.8	280	11.6	--	--	40.5
E6	NA/ M/ O/ L/ TS	50.6	260	13.9	--	54.1	--
E7	NA/ M/ O/ L/ TS	53.1	283	10.2	--	--	**
E8	NA/ M/ O/ -/ TS	49.9	254	14.2	--	59.6	--
E9	NA/ M/ O/ -/ TS	53.0	282	11.2	--	58.0	--
E10	NA/ M/ O/ -/ TS	52.8	280	--	15.6	--	**

L E G E N D

S/N = Sample Number

PC = Processing Code

- NA = Normalized @ 1750F (1 hour), and Austenitized @ 1650F (0.5 hour).
- M = Marquenched @ 1000F (0.5 hour).
- O = Oil Quenched to Room Temperature
- L = Liquid Nitrogen Quenched
- TS = Initial Temper @ 600F (2 + 2 hours), and Stress Relieved @ 550F (1 hour).

HRC = Hardness, Rockwell "C" Scale

UTS = Ultimate Tensile Strength (ksi)

KIhem = Hydrogen embrittlement or hydrogen assisted stress cracking, Stress Intensity threshold; ksi.SQR(in). Testing @ -1.2V vs SCE in 3.5% NaCl Solution.

KIscc = Stress corrosion cracking, open circuit Stress Intensity threshold; ksi.SQR(in). Testing @ -0.6V vs SCE in 3.5% NaCl Solution.

KIc = Fracture Toughness per ASTM E399; ksi.SQR(in).

KId = Dynamic Fracture Toughness; ksi.SQR(in).

** = Data not obtained; invalid test.

4.4 <u>Tensile Strength Summary</u>: Table 3 summarizes the tensile properties from published sources for comparison to the results of the average ultimate tensile strength of the five steels after heat treatment, as determined from the HRC-correlation to UTS of FIG 3.

Table 8

Summary of published data mechanical properties for ESR 4340 type steels.

STEEL	COMMENTS	YS	UTS
S/N A	HT #3710046	-	**289**
4340	S/N A 5 IN. SQ.	236	273
4340	S/N A 8 IN. SQ.	227	268
4340	S/N A 5 IN. X 12 IN.	232	269
4340	S/N A 2.5 IN. DIA.	234	276
S/N B	HT #N264-1R-1	-	**259**
4340V	AVG. 13 PROD. HEATS	223	265
4340V	MIL-S 8844C CLASS 1	217	260
S/N C	HT #N848-4R-1	-	**285**
4340M	6" x 12" FIRST HEAT	236	284
4340M	6" RCS FIRST HEAT	235	281
4340M	VAR HT #3831573	248	294
4340M	MIL-S-8844C CLASS 3	230	280
S/N D	HT #3710234	-	**287**
HP310	S/N D METTEK PROCESS.	256	302
HP310	VAR HT #3811931, AVG.	257	304
HP310	VAR HT #3811931, AVG.	263	310
HP310	L-dir TARGET VALUES	260/270	300/310
S/N E	HT #791-2R-3-1	-	**269**
D6ac	12" RCS FIRST HEAT	250	290
D6ac	12" RCS SECOND HEAT	264	294
D6ac	SPEC. B303 11947 B	240	---

UTS values of S/N's are from hardness correlation.
S/N E was lower than expected from Heat Treatment.
S/N D was overtempered in order to reach 53$\pm$ 1HRC range.
S/N A,C,D attained the intended hardness range.

Since the hardness was the only measure of tensile strength, a correlation was established using the data from three sources and a best fit curve was derived using the BAC correlation, which fit two of three empirical correlations of tensile strength (UTS) to the measured hardness. A plot of the hardness relationships is shown in FIG. 3. The target hardness range of 53 ± 1 HRC or 52 to 54 HRC is seen to correspond to 280 ± 10ksi.

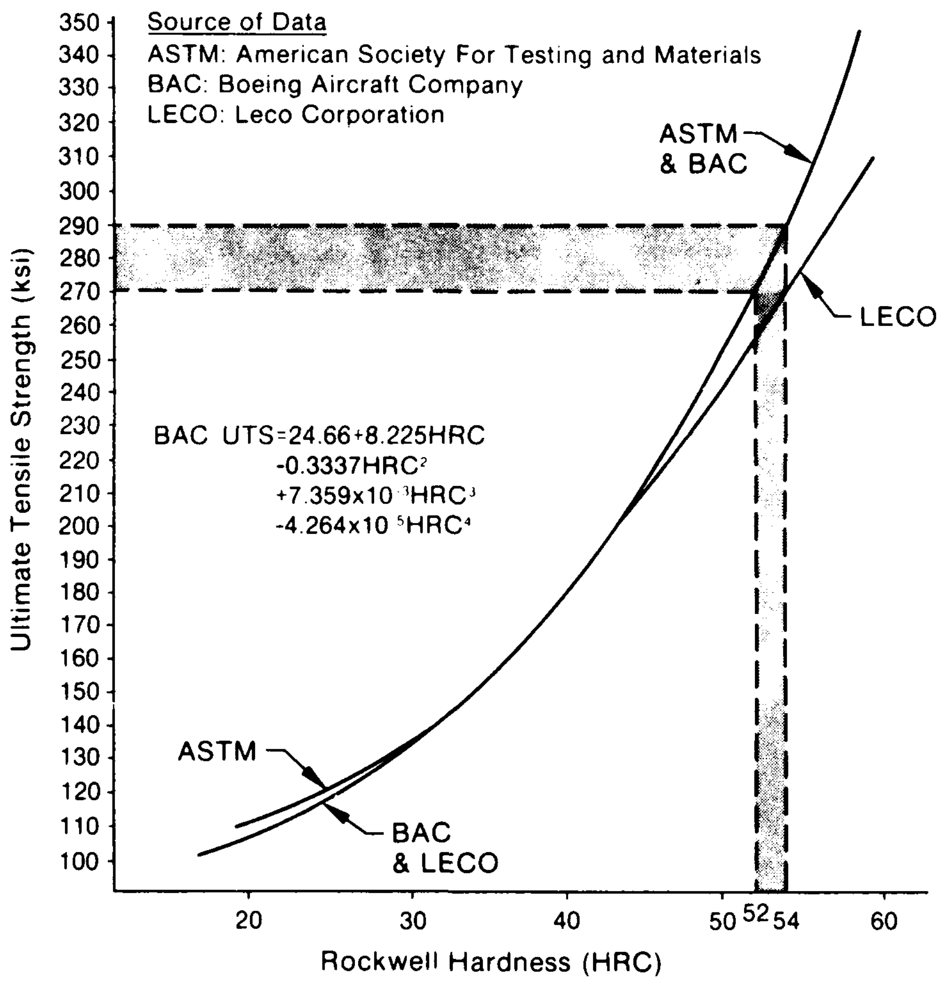


FIG. 3. Correlation of ultimate tensile strength to HRC.

4.5 KIhem as a function of heat treatment: Table 9 summarizes there results and the average KIhem measurements for the two groups of alloy steels (silicon additions or carbide former additions) and the four heat treatment modifications.

Table 9

KIhem as Function of Alloying Elements and Heat Treatment

STEEL		HEAT TREATMENT				AVG
TYPE	(Si)	STD	+ LN2	MAR	+ LN2	
4340	(.25)	11.1	09.7	11.4	12.4	
		10.7	11.0	10.8	11.6	
		10.3	--	10.6	--	11.0
9.1 Effect of Silicon Additions						
4340M	(1.5)	12.8	14.0	12.7	13.3	
		11.9	15.2	15.3	15.9	13.9
HP310	(2.5)	13.8	13.3	12.6	11.7	
		12.7	13.3	12.4	12.6	
		12.7	--	12.5	--	12.8
9.2 Effect of Carbide Formers						
4340V	(.1V)	15.8	13.3	15.5	18.0	
		13.5	12.5	12.5	14.4	14.4
D6Ac	(*)	12.3	14.0	14.2	13.9	
		11.6	10.7	11.2	10.2	12.3

* = Increased Chromium and Molybdenum; decreased Nickel. Vanadium maintained at 0.1 percent.

The individual data points of Table 9 are plotted in FIG. 4.

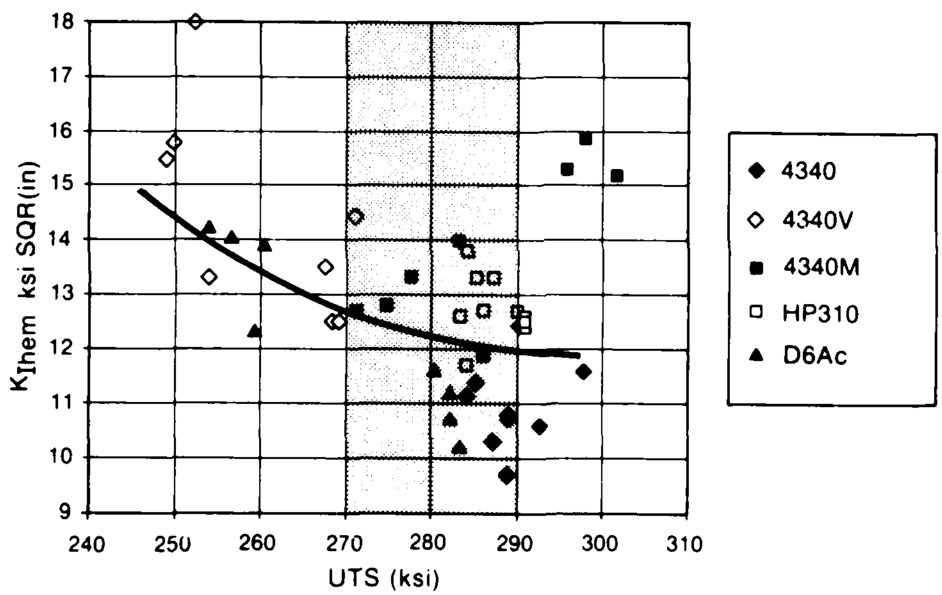


FIG. 4 Threshold KIhem vs. Ultimate Tensile Strength.
The target tensile range is shown in shaded area.

4.6 KIc as a function of heat treatment: Table 10 summarizes the results and the average KIc measurements for the two groups of alloy steels and the four heat treatment modifications.

Table 10

KIc as Function of Alloying Elements and Heat Treatment

STEEL TYPE	(Si)	HEAT TREATMENT STD	+ LN2	MAR	+ LN2	AVG
4340	(.25)	45.8	44.8	48.4	43.7	
		44.2	--	47.7	--	45.8
10.1 Effect of Silicon Additions						
4340M	(1.5)	48.5	49.0	49.0	46.5	
		--	--	48.7	--	48.3
HP310	(2.5)	46.0	37.7	48.2	39.2	
		44.7	--	48.0	--	44.0
10.2 Effect of Carbide Formers						
4340V	(.1V)	71.1	63.6	72.1	69.8	
		--	--	84.4	--	72.2
D6Ac	(*)	53.5	53.5	59.6	54.1	
		55.2	--	58.0	--	55.7

* = Increased Chromium and Molybdenum; decreased Nickel. Vanadium maintained at 0.1 percent.

The average value of the KIc and KIhem are compared in FIG. 5. Superimposing the tensile strength measurement shows the high-toughness and high-SCC or HEM resistance to be related to the lowest-strength 4340V alloy steel.

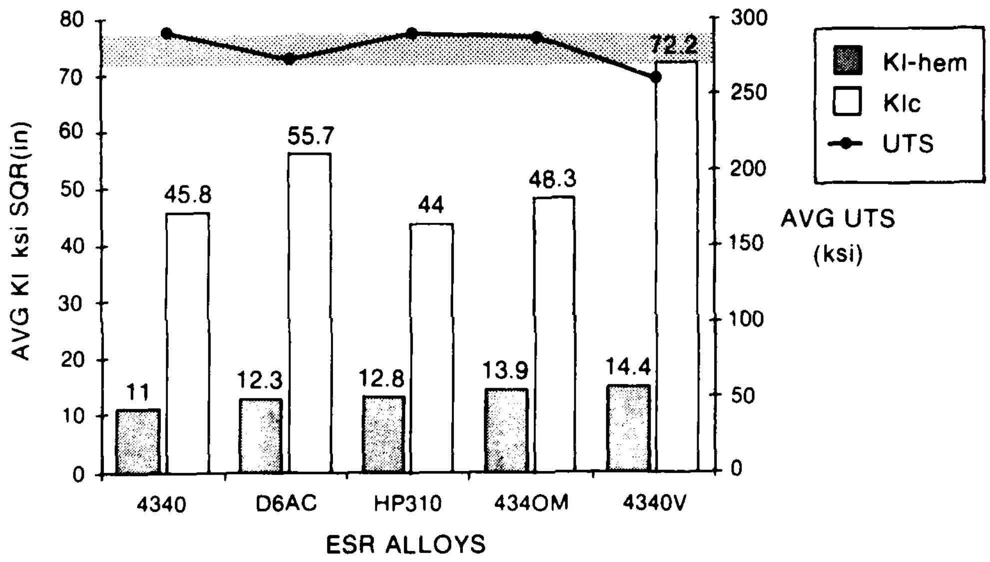


FIG. 5. An overlay of tensile strength to illustrate the sensitivity of the KIc and KIhem results to the strength. The results are positioned to rank the steels in order of increasing resistance to HEM from left to right.

4.7 KId as a function of heat treatment: Table 11 summarizes these results and the average KId measurements for the two groups of alloy steels and the four heat treatment modifications.

Table 11

KId as Function of Alloying Elements and Heat Treatment

STEEL		HEAT TREATMENT				AVG
TYPE	(Si)	STD	+ LN2	MAR	+ LN2	
4340	(.25)	46.5*	43.4*	41.6*	42.9*	43.6*
11.1 Effect of Silicon Additions						
4340M	(1.5)	38.3	31.4	--	--	34.9
HP310	(2.5)	39.8*	36.9*	39.6*	30.8*	36.8*
11.2 Effect of Carbide Formers						
4340V	(.1V)	48.4	45.5	48.8	50.4	48.3
D6Ac	(**)	40.5	36.4	--	--	38.5

* = Calculation based on estimate of load.
** = Increased Chromium and Molybdenum; decreased Nickel. Vanadium maintained at 0.1 percent.

4.8 KIscc vs KIhem: As stated previously, the threshold stress intensity for stress corrosion cracking is conventionally defined as KIscc. Testing is conducted under open circuit potential, which is about -0.6V vs SCE in a 3.5% salt water solution. Since the tests in this program were conducted under a hydrogen producing potential of -1.2V vs SCE, they were designated KIhem. As a special evaluation, the effect of corrosion potential on the test results was studied on selected test samples. The results are shown in Table 12 and plotted in FIG. 6:

Table 12

Effect of potential on threshold stress intensity

STEEL	KIhem	KIscc	Ratio
4340M-STD (1.5Si)	12.3	36.5	0.34
4340M-MAR (1.5Si)	14.0	31.0	0.45
4340V-STD (+0.1V)	14.7	37.7	0.39
D6Ac-STD (+CrMo-Ni)	12.0	12.4	0.97
D6Ac-MAR (+CrMo-Ni)	12.7	15.6	0.81

The conclusion from this small study is that significant differences do exist for some low alloy steels as a function of the corrosion potential. Based on the limited data, an alloy steel might have a higher threshold for stress corrosion cracking under open circuit potential (KIscc) than in a hydrogen producing environment (KIhem), which represents worst case hydrogen charging conditions such as those that occur during electroplating.

FIG. 6 is a plot of all of the test results, Table 3 through Table 7. Including both KIhem and KIscc data of Table 12. Superimposed is the trend line (TL) maximum and TL minimum taken from the ratio analysis diagram, RAD (Ref.5). Interestingly, the variation in results of this program encompasses the min-max range shown in the RAD. This observation suggests that the scatter in the RAD to a large degree, reflect differences in test methods. It should be noted that TLmax for SCC from the RAD coincides with the KIscc or open circuit (-0.6V vs SCE) test results; whereas, TLmin for SCC from the RAD coincides with the KIhem or -1.2V vs SCE test results.

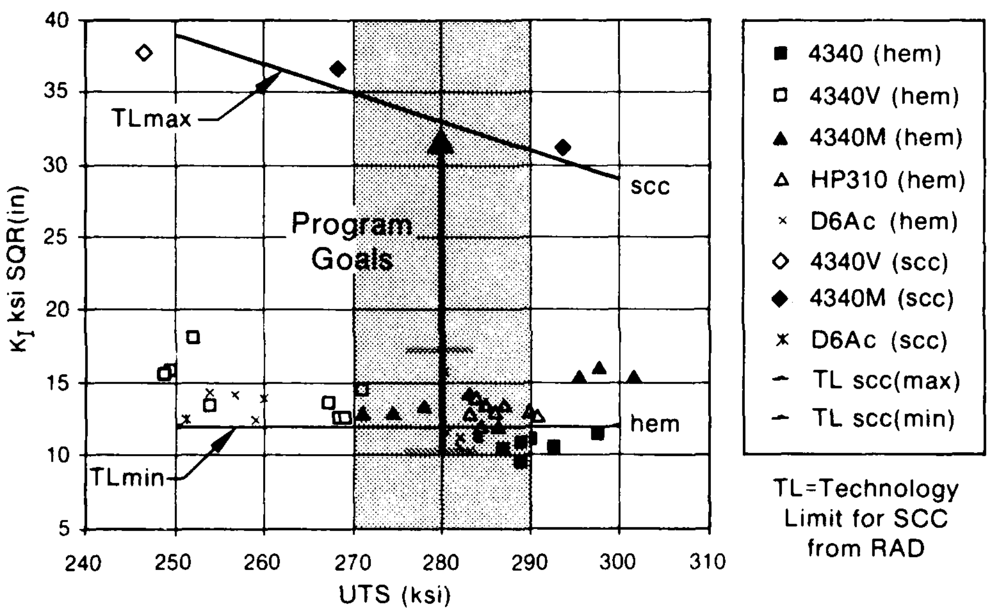


FIG. 6 KIhem & KIscc vs Ultimate Tensile Strength. The shaded area identifies the target tensile strength range. The program goal is to increase KIscc from a low value of about 10 ksi.SQR(in) to TLmax. The horizontal bar at 17ksi.SQR(in) reflects the maximim achievements of this program.

5.0 SUMMARY

An increasing number of hydrogen embrittlement failures in ESR 4340 steel at about 53HRC has created a need to improve the resistance of this alloy steel to hydrogen stress cracking. This program was designed to illustrate the effectiveness of alloying and heat treatment on commercial alloys (all ESR) that were selected to represent one group of steels with increasing silicon content and another group with increasing carbide formers. The basis for this selection was the contention that alloying can raise the tempering temperature required to produce a given hardness and thus provide more stress relief. This effect not found to be true as shown in FIG. 14, page 35. Above 500F, a decrease is observed.

The objective of this program was quantified by attempting to reach a threshold stress intensity level measured with vacuum arc remelted steels at the same hardness level; i.e., raise KIhem from 10 to 15ksi.SQR(in). From this point of view, success was only with 4340M @ 300ksi and D6Ac @250ksi UTS.

Results show that only slight modifications in chemistry, such as the addition of 0.1% vanadium, can significantly help to attain the desired goal. Use of 1.5% silicon (4340M, which also has 0.1%V) showed the best promise. Increasing the silicon to 2.5% (HP310) did not perform any better, although both alloy steels performed better than those with additional carbide formers. The gains with the carbide formers were usually at a sacrifice in strength. The suggestion is that the strength is a more significant variable than the use of a higher tempering temperature.

This program represents a rather exhaustive study that clearly illustrates the difficulty in generating a major advancement such as raising the threshold level to 30-40ksi.SQR(in) as with ESR or VAR 4340 steel at 43HRC. The program also clearly illustrates significant differences in stress corrosion testing under an open circuit potential (KIscc) as compared to testing in a cathodically charged hydrogen producing environment, KIhem, which is definitely more severe. This last observation suggests that caution be exercised in using stress corrosion test results for design purposes unless the test conditions are clearly scrutinized.

6.0 CONCLUSIONS

6.1. A large number of possibilities (five ESR type 4340 alloy steels and four heat treatments) were examined to illustrate that improvements in the resistance of ESR 4340 steel at 280 $\pm$10ksi ultimate tensile strength, to hydrogen embrittlement are small with conventional variations in heat treatment such as subzero cooling and marquenching. At best, KIhem increases to 15ksi.SQR(in) from 10ksi.SQR(in) are obtained. A threshold of fifteen corresponds to a value measured from a split heat of vacuum arc remelted steel at the same strength or hardness (53 $\pm$1HRC) level.

6.2. Silicon additions of 1.5% to ESR 4340 steel (300M or 4340M) are more effective than increasing carbide formers, especially if the strength is to be maintained. Additional amounts of silicon to 2.5% (HP310) did not add further improvement to KIscc at this lower strength level. Although HP310 steel was designed for use at 310ksi ultimate tensile and therefore should also be evaluated at this strength level.

6.3. No direct correlation to tempering temperature could be established. Similarly, no distinct advantage was found with marquenching to reduce the severity of the quench or varying the amount of retained austenite by subzero quenching. Although, in general, the least resistant condition of the alloy steels to hydrogen embrittlement appears to be with a conventional quench and temper heat treatment.

7.0 RECOMMENDATIONS

7.1. A more nonconventional approach must be used in modifying the heat treatment to generate significant improvements in the resistance of alloy steels to hydrogen stress cracking. A rapid austenite reversion step prior to quenching could be considered after an elevated temperature temper of about 1200F. In this way, the martensite might be changed from an acicular to a lathe structure. Information from the literature suggests that the lathe structure is more favorable with regard to inhibiting hydrogen assisted stress cracking.

7.2. The addition of 0.1% vanadium, commonly used for grain refinement, appears to have distinct advantages and should be used with ESR 4340 steels heat treated to hardnesses in excess of 53HRC. An additional 1.5% Si (4340M) improves tempering characteristics but does not significantly improve KIhem.

7.3. The use of a combination of both an interrupted quench and subzero cool could also be employed to maximize the resistance to hydrogen stress cracking, but from practical considerations this recommendation has restrictions in addition to being a secondary consideration compared to the vanadium and/or silicon addition.

8.0 SPECIAL ASSIGNMENTS

8.1. Heat treatment of HP310: The as-supplied HP310 ingot (HT #3710234) was difficult to machine because of its high-hardness, therefore, a separate assignment was to develop a heat treatment that would lower the hardness. The objective was met by a treatment that consisted of normalizing and tempering as follows:

Normalize	2hr @ 1775max, Air Cool
Temper	2hr @ 1275max, Air Cool
Temper	2hr @ 1250max, Air Cool

The resultant hardness was 40HRC, which could then be machined.

Hardening: After rough machining a tensile bar, the specimen was hardened to 54-55HRC as follows:

Austenitize	2hr @ 1600F, Air Cool
Temper	2hr @ 575F, Air Cool
Temper	2hr @ 575F, Air Cool

Note: Since this treatment was developed, an additional subzero cool for 2hr at -110F also has been incorporated after austenitizing in order to insure complete transformation to martensite.

Mechanical Properties: The tensile specimen was then finish machined and tested per ASTM E8 to record the following mechanical properties:

	Yield/Tensile	El/RA(%)
S/N D	255.6/301.8	10/39.8
Target	260-270/300-310	9-12/30-40

Conclusions: The usefulness of the ESR HP310 plate material supplied by MTL was established with a small test program prior to proceeding with the main test program. It was established that the billet must be normalized and tempered before machining and prior to heat treatment in order to obtain the target properties.

8.2 Use of KId/KIc ratio as index of susceptibility: Because of the tendency for intergranular fracture when an alloy steel is in the tempered martensite embrittlement region, the ratio of dynamic to conventional fracture toughness was evaluated as a means of identifying increased susceptibility to hydrogen embrittlement. Initially it was conceived that the toughness ratio would be less than unity if the steels were tempered in the embrittlement region or equivalent to 550F embrittlement for 4340 steel; otherwise, the ratio would be unity. Generally, the ratio should be unity when comparing dynamic to conventional fracture toughness for steels in excess of 140ksi.

Since tempering was only at one temperature, the comparative results could not be obtained with the scope of this program, but the measured results were used to generate the data plotted in FIG. 7 and FIG. 8. The results show a large amount of scatter, with the general trend that the ratio decreases with increased resistance to hydrogen stress cracking (FIG. 7) and increases with UTS (FIG. 8). This effect is opposite to what might be expected, thereby introducing a lot of uncertainty as to the interpretation of the results. Obviously, insufficient test data exists and more testing must be performed before any substantiated conclusions can be reached.

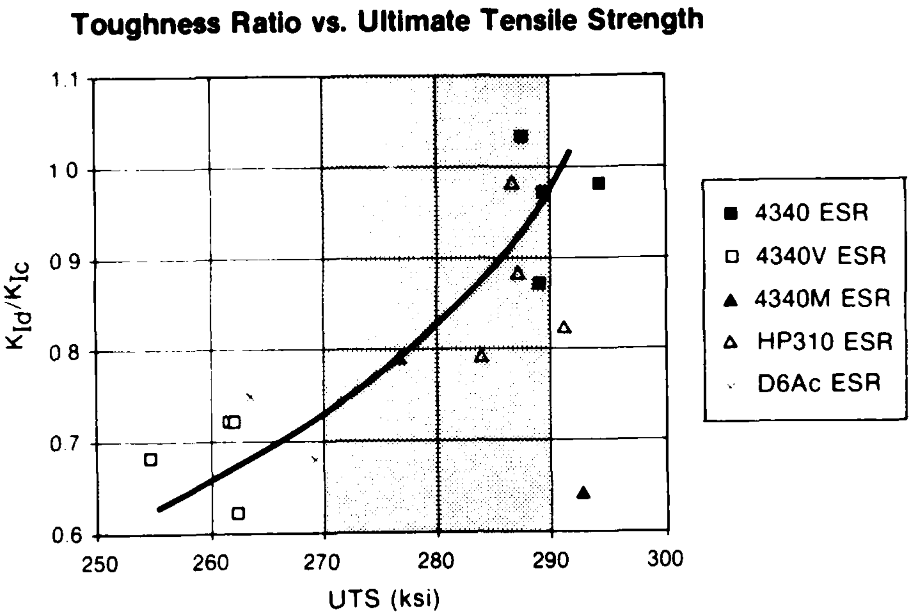


FIG. 7 Toughness ratio vs KIhem

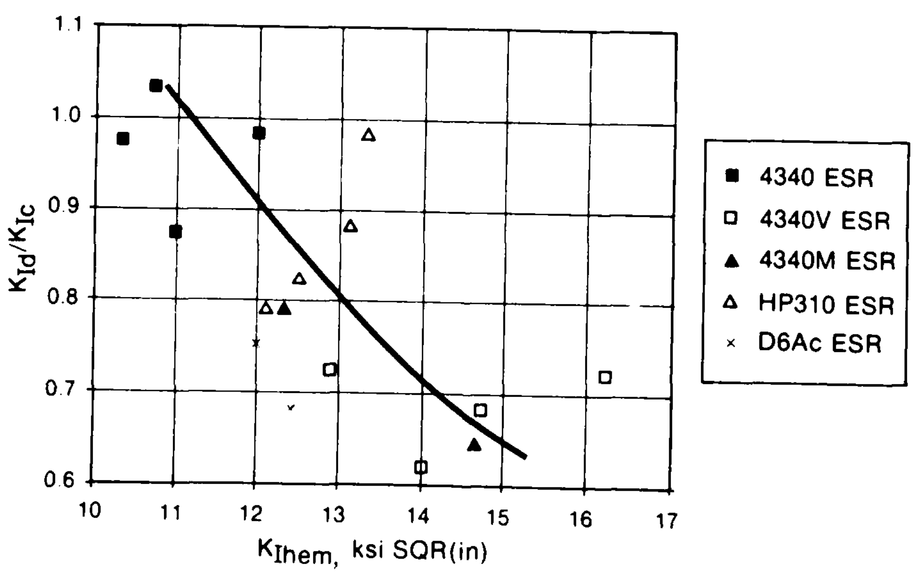


FIG. 8 Toughness ratio vs UTS

8.3 Ratio Analysis: The ratio of the critical stress intensity for fracture (KIc) or for sustained load subcritical crack growth in an environment (KIscc) to the yield strength (YS) is an index of damage tolerance (DTI) because it is related to a critical crack size for fracture or environmentally assisted cracking. In turn, the DTI-ratio is related to the level of nondestructive testing required for quality assurance. As an example, for a center cracked panel the critical crack size (a_c) is given by:

$$a_c = 0.25 \circ FS^2 \circ \text{DTI-ratio}^2$$

where FS = Factor of Safety or Yield Strength divided by the applied stress.

and DTI-ratio* = KIc or KIscc/ Yield Strength (YS)

Obviously, as the factor of safety or DTI-ratio increase, the critical crack size increases, both for fracture, when KIc is used or for environmentally assisted cracking, when KIscc is used in the above equation.

The material technology limits are defined by the ratio analysis (RAD) diagram shown in FIG 9, where both the KIscc zoning is overlayed on the fracture transition technology limit (TL-F). Also identified is TL-scc limit.

The region of interest is above 200ksi yield, or a zone, intersecting with a DTI-ratio for fracture at unity or KIc = YS. This region is magnified in FIG 10. The following information can be extracted from FIG 10 if the estimate that YS + 40ksi = TS (Tensile Strength) is used.

CRITERIA	UTS => / YS =>	240 / 200	260 / 220	280 / 240	300 ksi / 260 ksi	
Fracture (TL-F)		1.0	0.7	0.5	0.4	SQR(in)
KIc DTI-ratio		31.6	22.1	15.8	12.6	SQR(mil)
(TL-scc)		0.7	0.3	0.15	0.12	SQR(in)
Env DTI-ratio		22.1	9.5	4.7	3.8	SQR(mil)
KIscc/KIc (TL-scc)/(TL-F)		70	43	30	30	%

* For 100% structural integrity, a proof test is required when $a_c \leq .050$in or DTI $\geq$ 1 / (2FS).

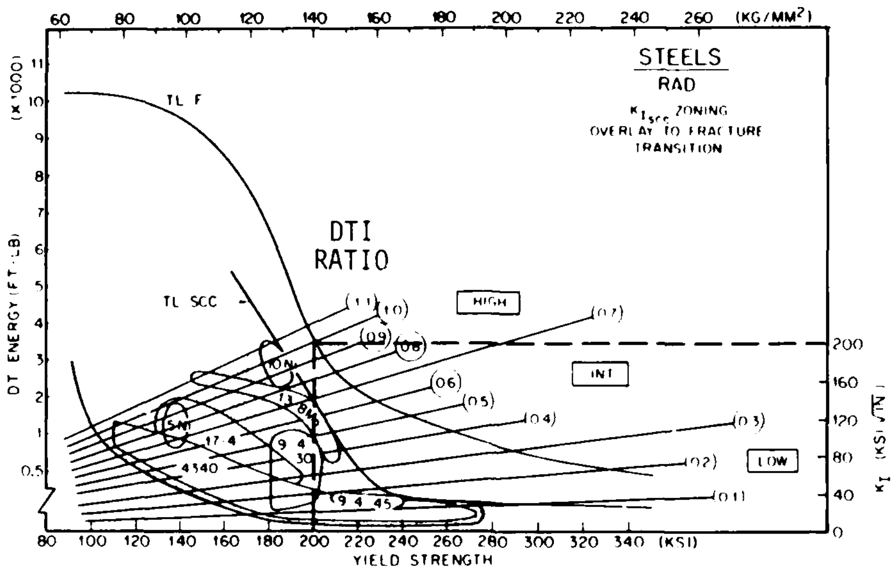


FIG. 9. RAD fracture diagram for steels with KIscc overlay.

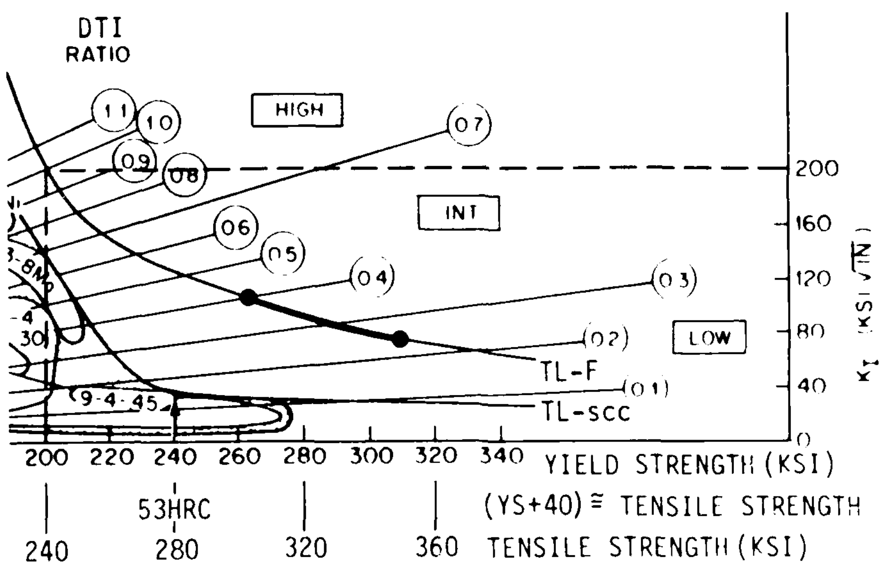


FIG. 10. Enlargement of RAD for high-strength steels to illustrate program goals.

8.4 DTI-ratio: Using the previous Table or FIG 9 or FIG 10 as a guideline to give perspective to the program and establish target properties, the following comparisons can be made with regard properties measured on the ESR steels tested in this program.

8.4.1 Fracture: With regard to fracture toughness, proposed research programs on innovations to high-strength steel technology have cited DTI-ratio goals of 120/300(TS) = 120/260(YS) = 0.4SQR(in) and 60/350(TS) = 60/310(YS) = 0.2SQR(in). These goals are consistent with the RAD for they fall on the TL-Fmax curve as shown by the bold line on FIG 10.

Our target tensile properties are 280 $\pm$10ksi or about 240ksi yield (YS), converted from 53 $\pm$1HRC. From the RAD, the maximum DTI-ratio for fracture is about 0.5SQR(in) at TL-Fmax. The measured properties are nominally 50/240 or about 0.2SQR(in) or 40% TL-Fmax FIG 11 is a plot of all measured test results.

8.4.2 Stress Corrosion: With regard to stress corrosion or in our case, hydrogen embrittlement, the target DTI-ratio is 0.15SQR(in) or 4.7SQR(mil) from the RAD TLmax-scc curve. The measured properties averaged 0.05 to 0.06SQR(in) or 1.3 to 2.0SQR(mil) or about 35% TL-SCCmax. FIG 12 is a plot of all measured test results.

8.4.3 %KIc: With regard to the ratio of (KIscc or KIhem)/KIc, a target value is 30% based on TLmax values. The measured properties are about 24% for ESR 4340, 21% for carbide formers, and 30% for silicon additions, primarily reflecting variations in KIc.

The conclusion from these observations is that there is room for improvement in raising KIscc or KIhem to TL-SCCmax, and also in raising KIc to TL-Fmax. Focusing on alloy and heat treatment modifications that improve fracture toughness will not necessarily produce concomitant increases in resistance to hydrogen assisted stress cracking.

The DTI-ratio from this program for both SCC and fracture is compared in FIG 12 to the test results from a previous program, using a split heat of 4340 steel with each half subsequently remelted either by electroslag (ESR) or vacuum arc (VAR). As noted, only near the 300ksi tensile strength range do the alloy and heat treat modification from this program show any beneficial advantages.

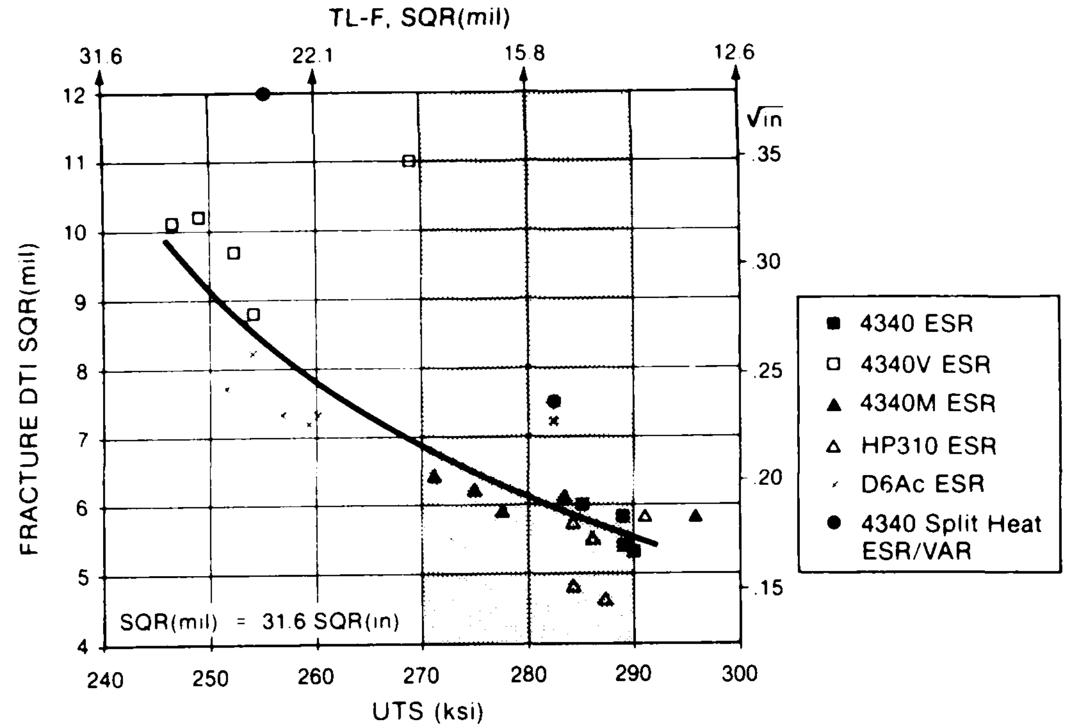


FIG. 11 Average fracture DTI-ratio vs Ultimate Tensile Strength.

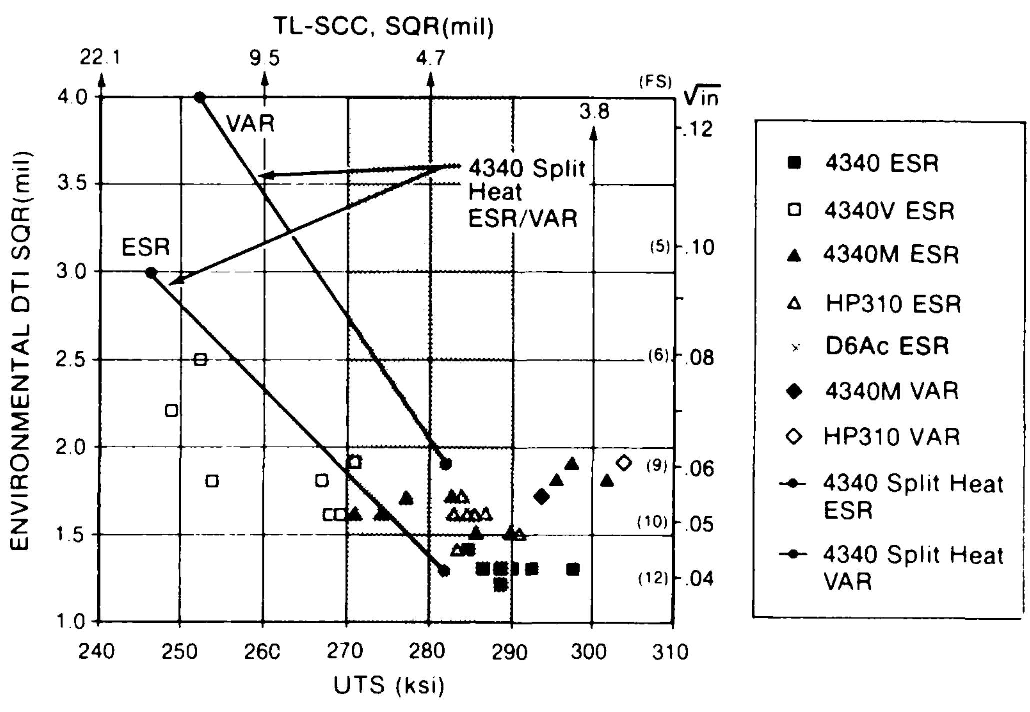
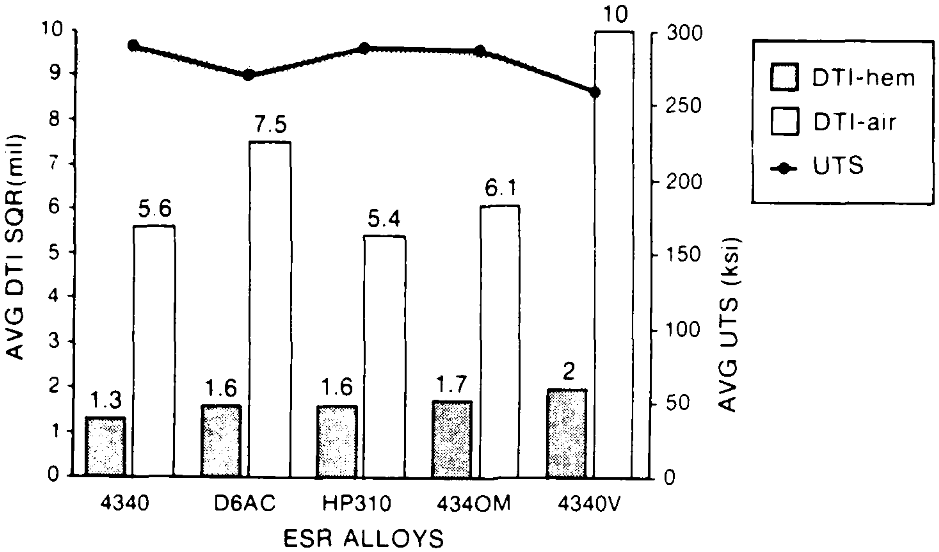


FIG. 12 Average environmental DTI-ratio vs Ultimate Tensile Strength.



* DTI in units of SQR(mil) = ($10^{1.5}$) SQR(in) = 31.6 SQR(in)

FIG. 13. Average DTI-ratio & UTS for five ESR 4340 alloy steels positioned in order of increasing resistance to hydrogen stress cracking. The shaded band identifies the target tensile properties with 4340V slightly below minumum.

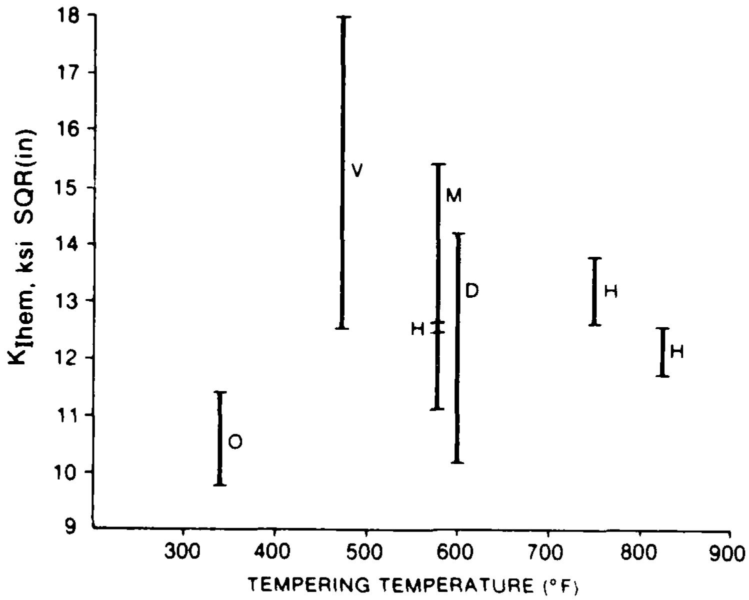


FIG. 14. Resistance to HSC shown not to increase with tempering temperature.

8.5 Presentation at TRI-SERVICES Conference on Corrosion: On invitation from MTL, an oral presentation was given at the Tri-Services Conference in Orlando, Florida on 2-5 December, 1985. (Ref 6.)

8.6 Publication in the Proceedings of TRI-SERVICES Conference on Corrosion: On invitation from MTL, a paper was written for publication in the proceedings of the Tri-Services Conference in Orlando, Florida on 2-5 December, 1985. (Ref 6.)

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2. Raymond, L., "Accelerated KIscc/KIc Testing," Proceedings of the Army Symposium on Solid Mechanics, 1982 - Critical Mechanics Problems in Systems Design, AMMRC MS 82-5, 1982.

3. Raymond, L., "Screening Test for Hydrogen Embrittlement," Proceeding International Symposium for Testing and Failure Analysis, 1981.

4. Venal, W.V., "ESR of Ultra High-Strength Steels," Proceeding of the 39th Electric Furnace Conference, 1981.

5. Pellini, W.S., Principles of Structural Integrity Technology, Office of Naval Research, p.188, 1976

6. Raymond, L., "Effects of Alloying Elements and Heat Treatment of Type 4340 ESR High-Strength Steels on Hydrogen Embrittlement", Presented and Published in the Proceeding of the Tri-Services Conference on Corrosion, 1985.

10.0 ACKNOWLEDGEMENT

The author expresses his appreciation for the cooperative effort put forth by the program monitor, A. A. Anctil. W. Hallerberg of the Ingersoll-Rand Company, who donated the three exploratory heats of ESR steels is also duly acknowledged.

APPENDIX

Documentation of test results

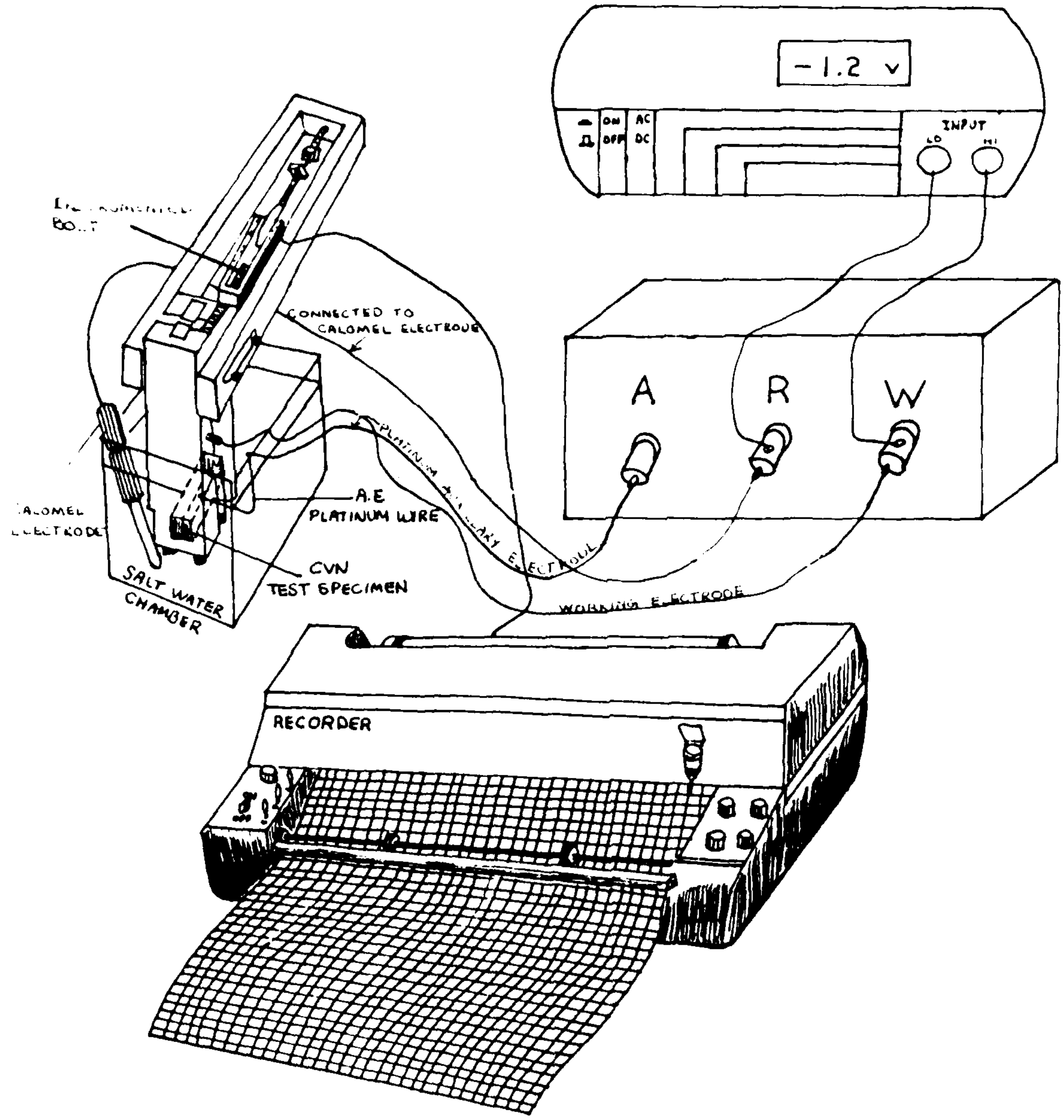
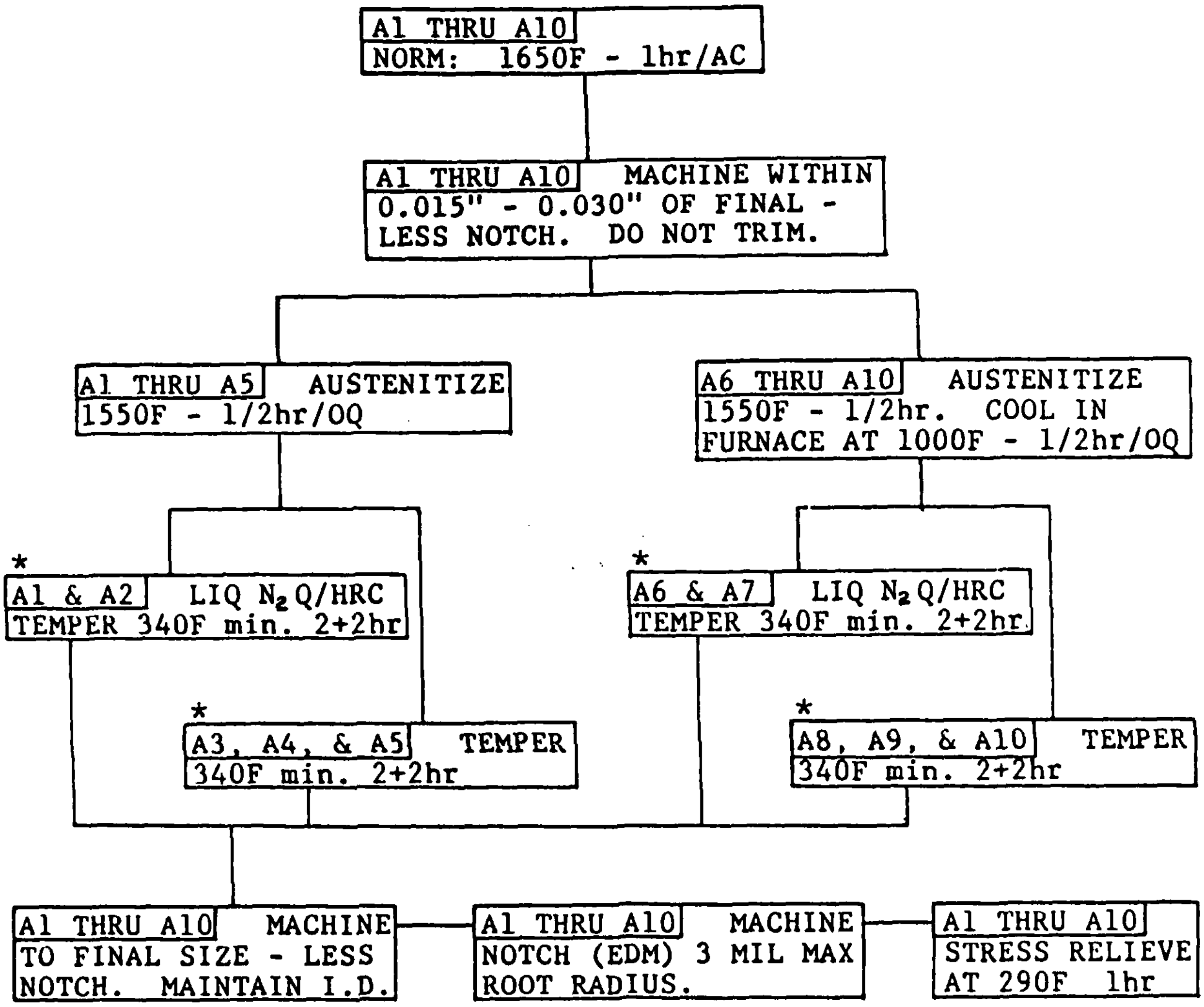
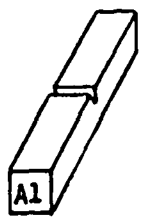


FIG A1.0 Schematic of RIM SCC-testing System.

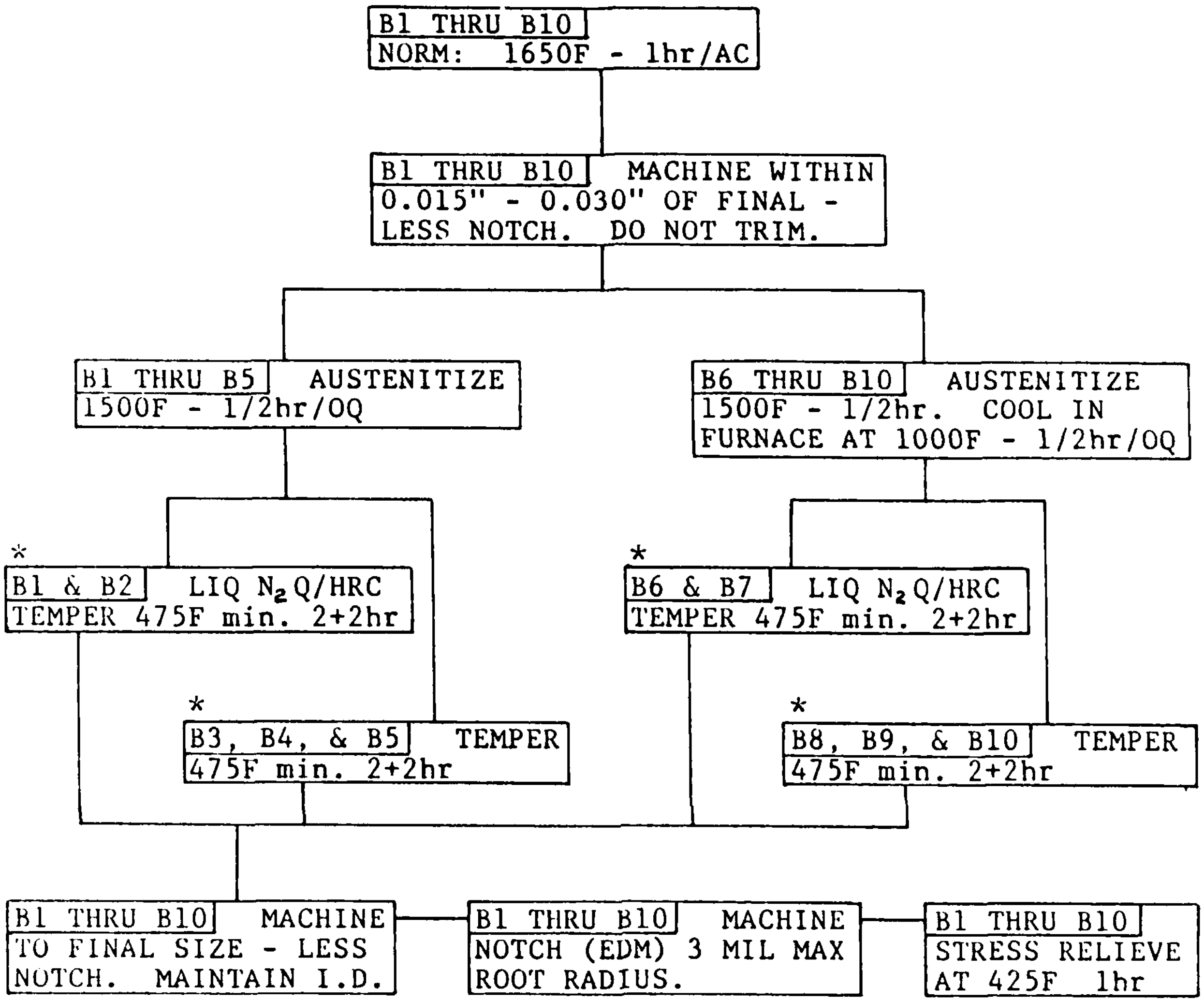


* ADJUST SECOND TEMPERING TEMPERATURE TO OBTAIN REQUIRED HARDNESS OF 52 - 54 HRC.

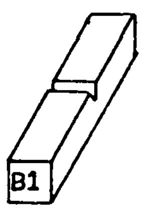


MAINTAIN STAMPED IDENTITY ON ONE END OF SPECIMEN AFTER FINAL MACHINING (A1 SHOWN AS EXAMPLE) ORIENT NOTCH AS SHOWN.

FIG. A2.1. Specimen preparation Flow Chart for S/N A: 4340

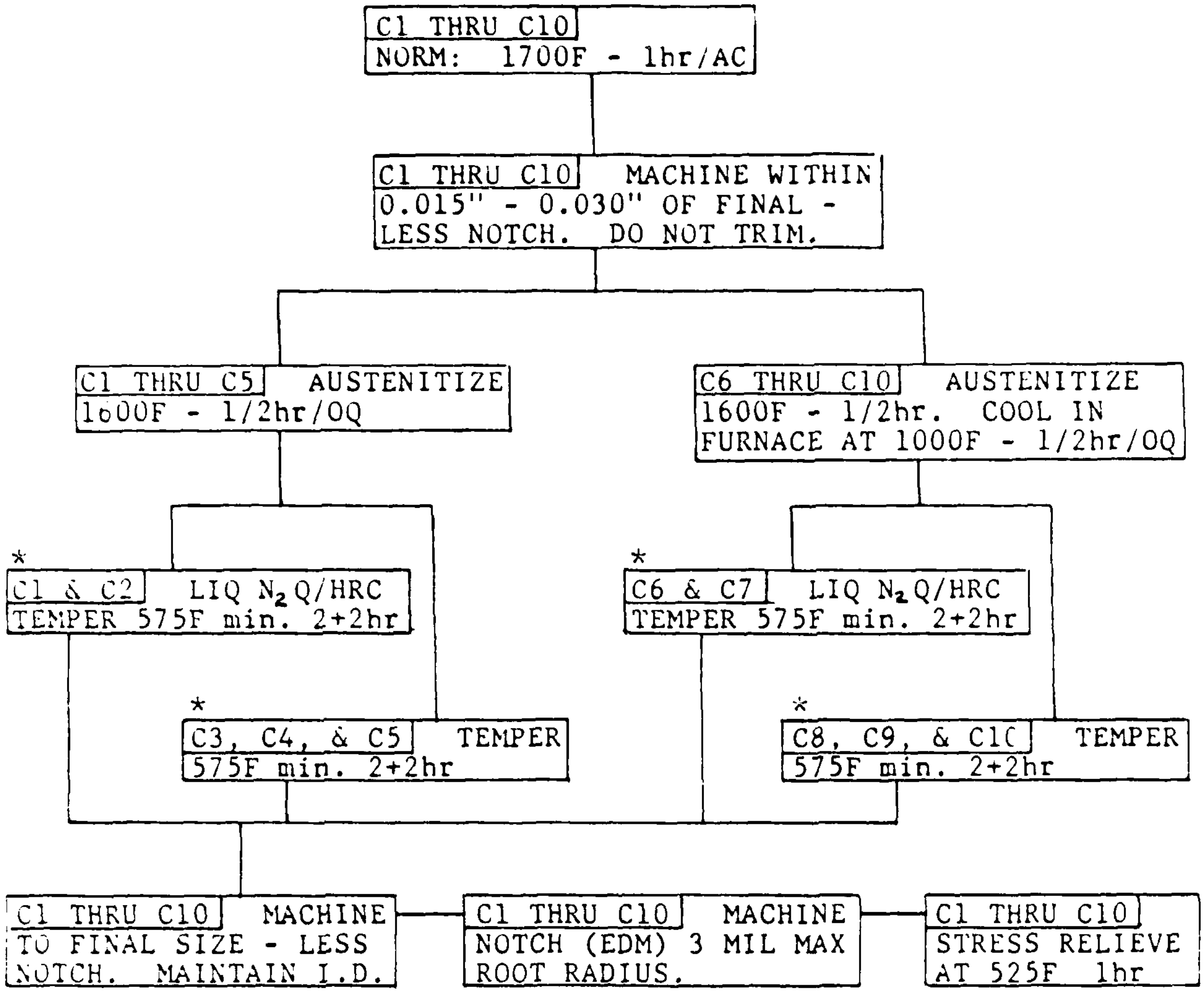


* ADJUST SECOND TEMPERING TEMPERATURE TO OBTAIN REQUIRED HARDNESS OF 52 - 54 HRC.

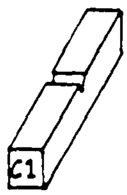


MAINTAIN STAMPED IDENTITY ON ONE END OF SPECIMEN AFTER FINAL MACHINING (B1 SHOWN AS EXAMPLE) ORIENT NOTCH AS SHOWN.

FIG. A2.2. Specimen preparation Flow Chart for S/N B: 4340V

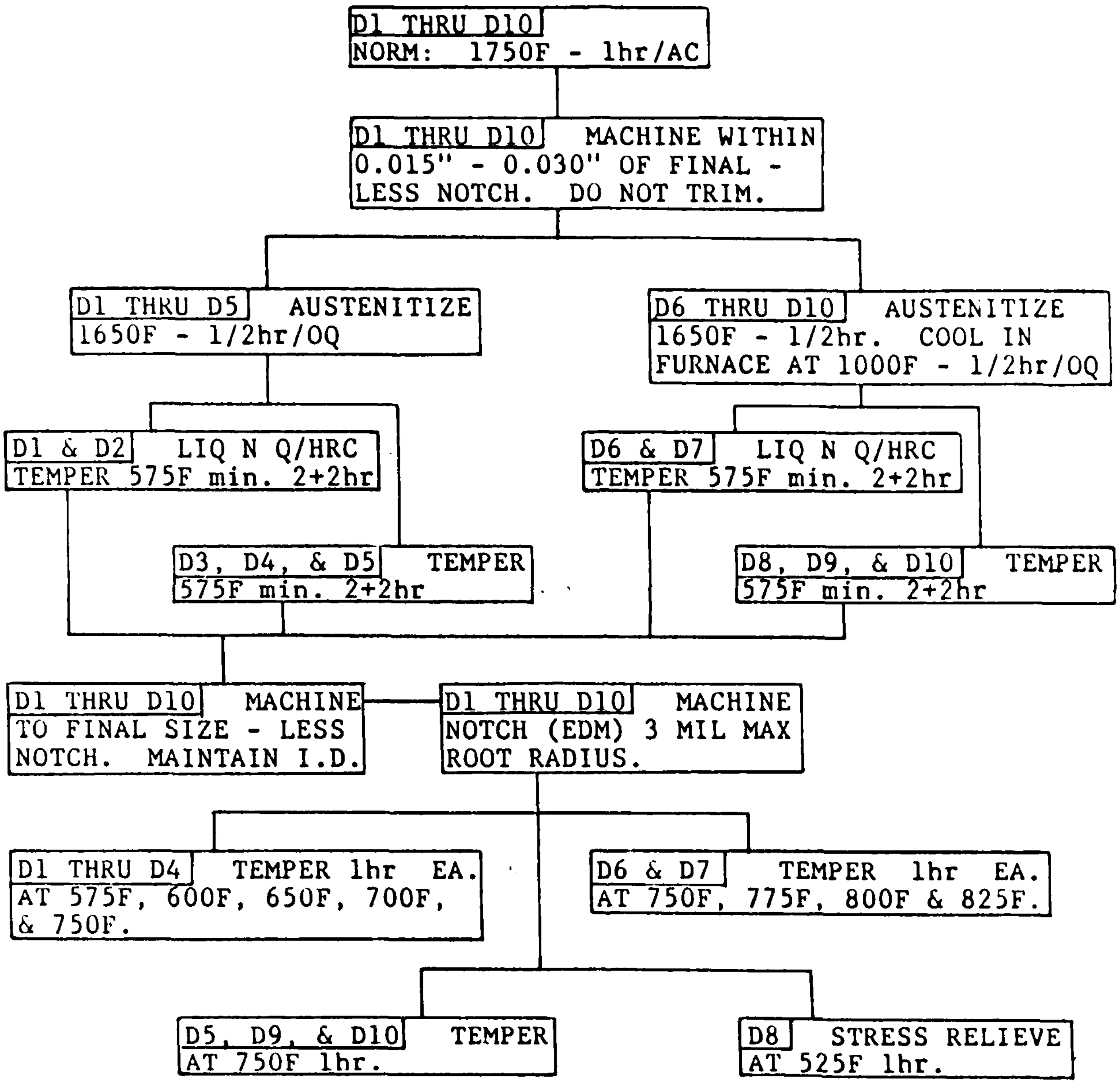


* ADJUST SECOND TEMPERING TEMPERATURE TO OBTAIN REQUIRED HARDNESS OF 52 - 54 HRC.

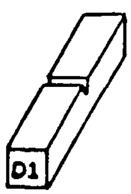


MAINTAIN STAMPED IDENTITY ON ONE END OF SPECIMEN AFTER FINAL MACHINING (C1 SHOWN AS EXAMPLE) ORIENT NOTCH AS SHOWN.

FIG. A2.3. Specimen preparation Flow Chart for S N C: 4340M

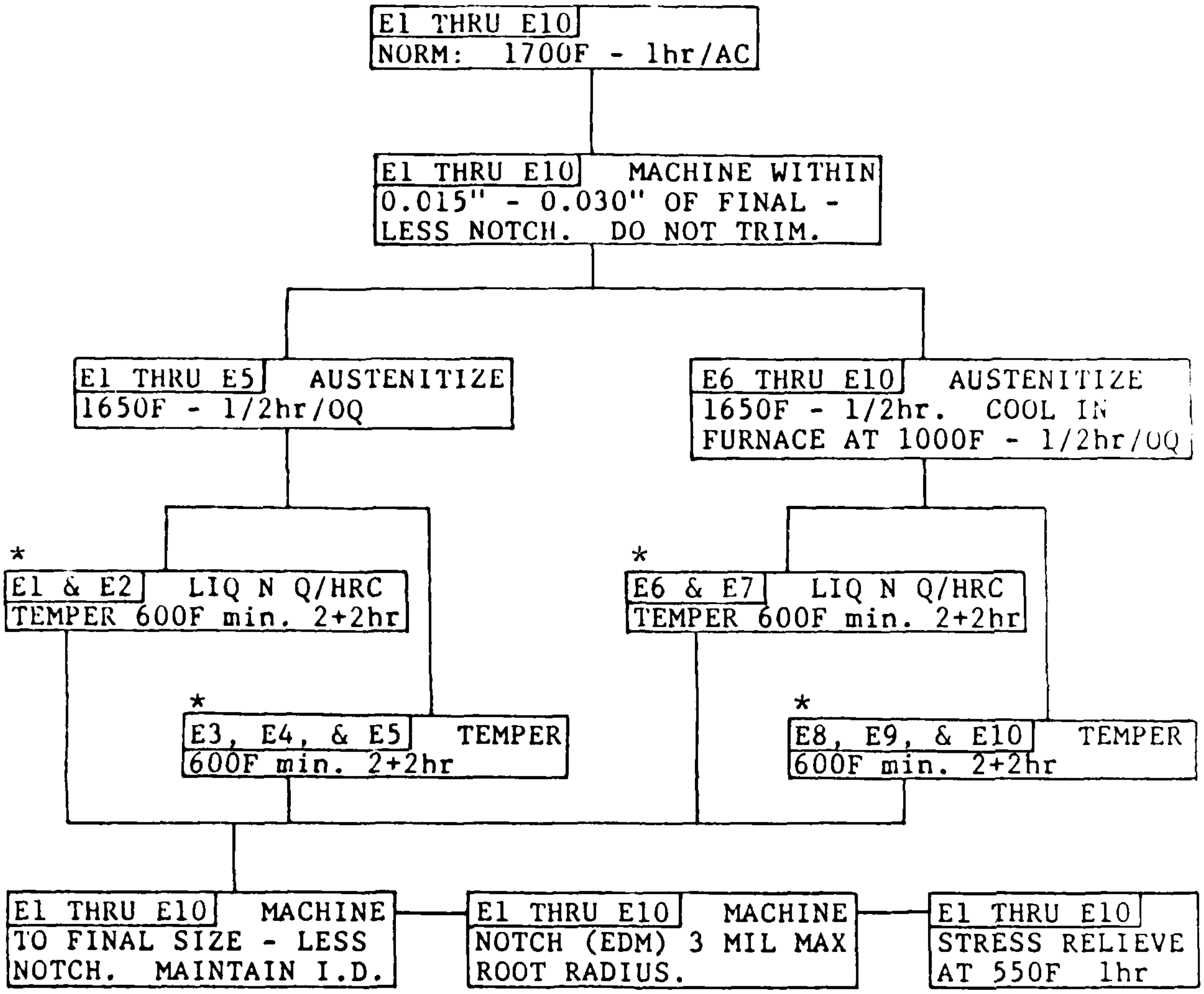


SPECIMENS TEMPERED TO OBTAIN REQUIRED HARDNESS OF 52-54 HRC.

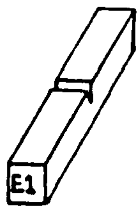


MAINTAIN STAMPED IDENTITY ON ONE END OF SPECIMEN AFTER FINAL MACHINING (D1 SHOWN AS EXAMPLE) ORIENT NOTCH AS SHOWN.

FIG. A2.4. Specimen preparation Flow Chart for S/N D: HP310

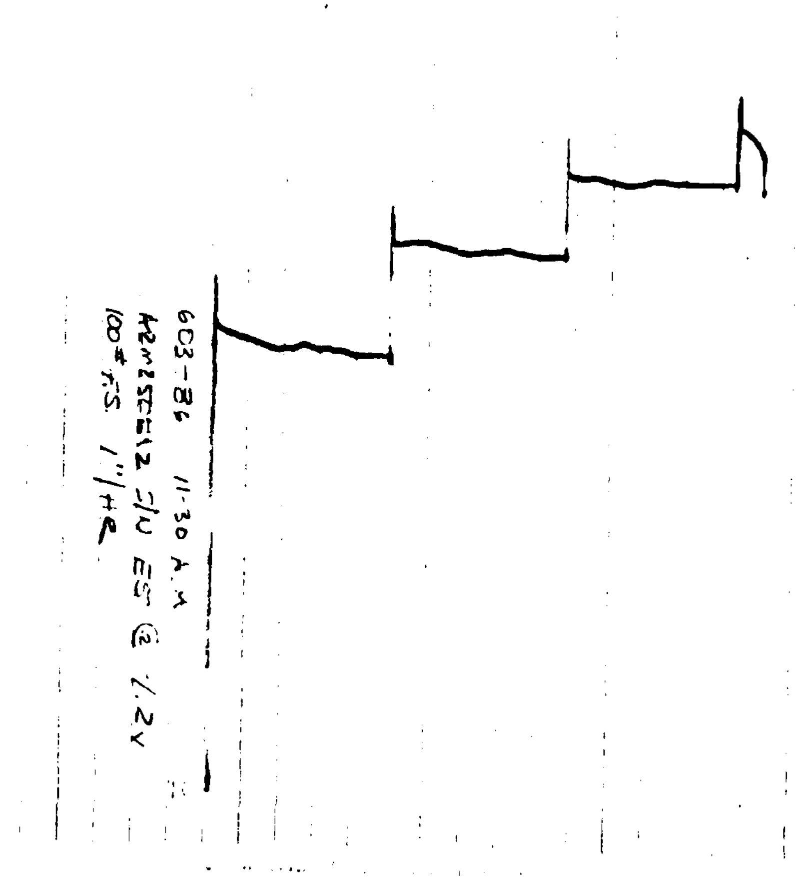


* ADJUST SECOND TEMPERING TEMPERATURE TO OBTAIN REQUIRED HARDNESS OF 52 - 54 HRC.



MAINTAIN STAMPED IDENTITY ON ONE END OF SPECIMEN AFTER FINAL MACHINING (E1 SHOWN AS EXAMPLE) ORIENT NOTCH AS SHOWN.

FIG. A2.5. Specimen preparation Flow Chart for S/N E: D6Ac



S/N E5 100 lbs FS 1"/HR @ -1.2v 6-03-86

FIG. A3. Example of step-load traces.

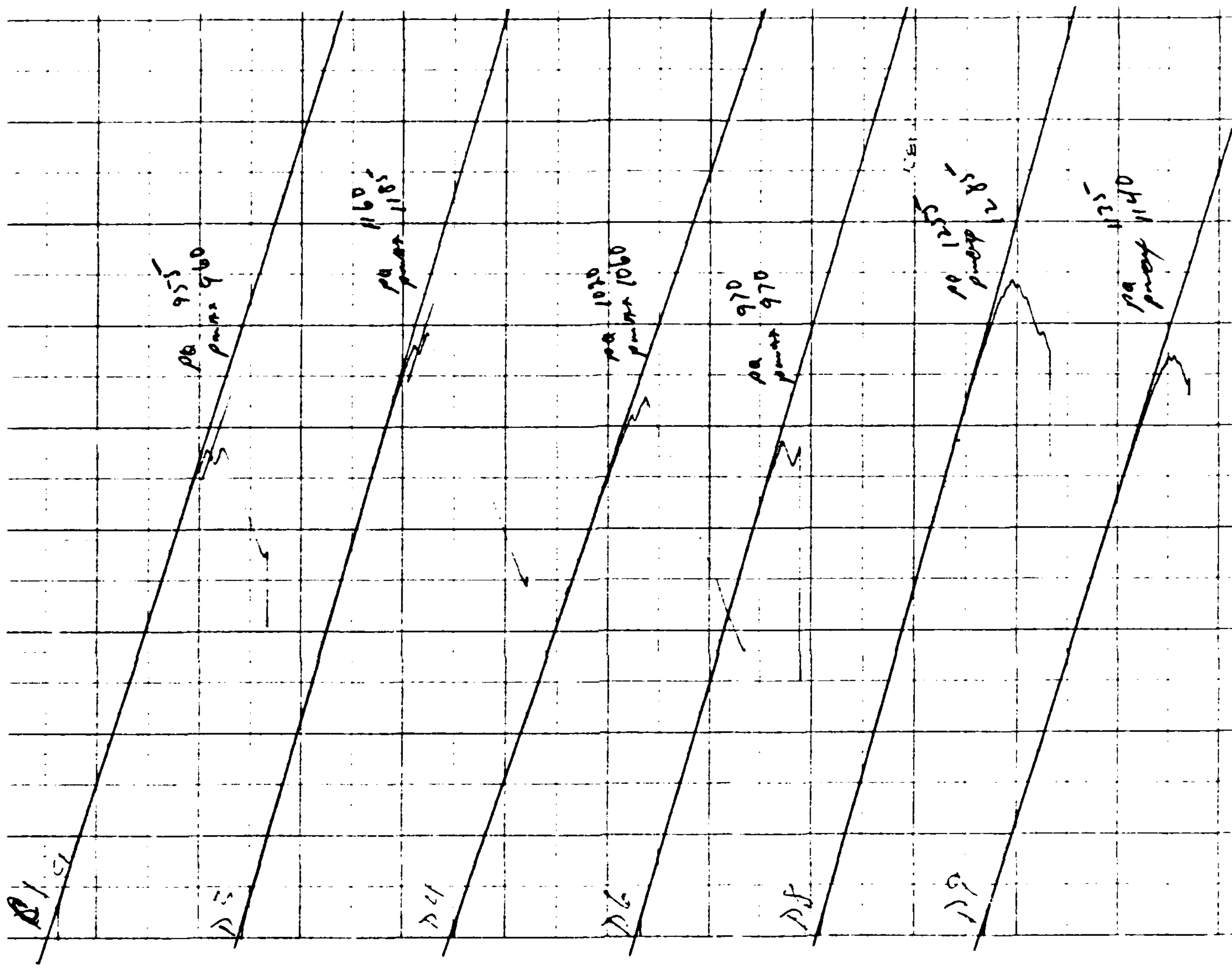


FIG. A4. Example of fracture toughness data.

IMPACT TEST DATA SHEET

TEST #: [illegible] DATE: [illegible]

WORK REQUEST #: ________ TECHNICIAN: ________

SPECIMEN DATA:

MATERIAL: ________ HARDNESS: ________ R_c LENGTH: ________ IN

TYPE: [illegible] NOTCH DEPTH: ________ IN WIDTH: ________ IN

FEATURES: [illegible] NOTCH RADIUS: ________ IN DEPTH: ________ IN

TEST RESULTS:

DIAL ENERGY: 15 FT-LB MAXIMUM LOAD: ________ LB

TOTAL ENERGY ($\triangle E_o$): ________ FT-LB % SHEAR: ________ %

INITIATION ENERGY: ________ FT-LB LATERAL EXPANSION: ________ IN

PROPOGATION ENERGY: ________ FT-LB

TEST DATA:

	Ea' (FT-LBS)	t (MSEC)	Ea = Ea' (1 + .008t)	$\triangle E_o = Ea\,(1 - \frac{Ea}{4Eo})$
TOTAL ENERGY				
INITIATION ENERGY				

PROPOGRATION ENERGY = (TOTAL ENERGY) - (INITIATION ENERGY)

TEST CONDITIONS:

AVAILABLE ENERGY (E_o): 264 FT-LB SPECIMEN TEMP.: 75 °F

HAMMER ANGLE: 120 DEG AMBIENT TEMP.: 75 °F

HAMMER VELOCITY: [illegible] CM/SEC

OSCILLOSCOPE SETTINGS:

SWEEP TIME: .1 MSEC/DIV DIAL VELOCITY: 512 CM/SEC

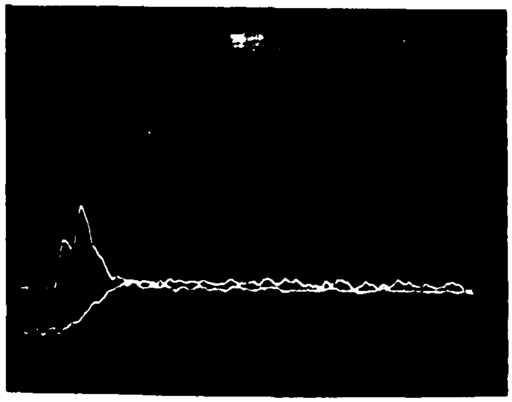
LOAD: 500 LB/DIV SCALE FACTOR: 1

ENERGY: 1 FT-LB/DIV FILTERS: Ⓐ IN OUT

Ⓑ IN OUT

TEST NOTES:

[illegible]



FOOTNOTES:

Ea' = ENERGY APPARENT FROM TRACE.

Ea = APPARENT ENERGY CORRECTED FOR CAPACITOR DECAY.

$\triangle E_o$ = ACTUAL ENERGY ABSORBED.

FIG. A5. Example of instrumented impact data.

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STUDY OF EFFECTS OF ALLOYING AND HEAT TREATMENT ON HYDROGEN EMBRITTLEMENT SUSCEPTIBILITY OF ELECTROSLAG REMELTED 4340 STEEL
Louis Raymond
L. Raymond & Associates
Irvine, CA 92715

Technical Report MTL TR 86-42, October 1986, 49 pp -
Illus-tables, Contract DAAG46-85-C-0020
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Final Report, 12/15/84 to 7/12/86

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This program focuses on the need to improve the resistance of high-strength steels to hydrogen embrittlement or hydrogen stress cracking. Variations in heat treatment and modifications in alloy composition of electroslag remelted 4340 steel at 53 HRC were extensively explored. Target goals were established in terms of a threshold stress intensity parameter, KIscc for open circuit potential conditions, designated KIhem for cathodic charging conditions under stress during test, which were the primary test conditions in this program. In general, all alloy additions improved KIhem. The addition of 0.1% vanadium appears to be the most significant individual modification to ESR 4340 steel in that it alone provides the same gains as the more heavily alloyed ESR 4340 steels. The results must be somewhat qualified because the hardness of ESR 4340V was 50 HRC instead of the intended 53 HRC. Silicon additions of about 1.5% tended to maximize the benefits from alloy modifications. Although within an alloy system the heat treatment effects were minor or secondary relative to alloy additions, the use of an intermediate quench and subzero cooling appeared to maximize the benefits from heat treatment. Increasing the threshhold KIhem was not directly related to tempering temperature as anticipated. Overall, KIhem was increased from 10 ksi SQR(in.) to a maximum value of about 15 ksi SQR(in.). Other improvements must be based on nonconventional approaches to thermal processing.

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This program focuses on the need to improve the resistance of high-strength steels to hydrogen embrittlement or hydrogen stress cracking. Variations in heat treatment and modifications in alloy composition of electroslag remelted 4340 steel at 53 HRC were extensively explored. Target goals were established in terms of a threshold stress intensity parameter, KIscc for open circuit potential conditions, designated KIhem for cathodic charging conditions under stress during test, which were the primary test conditions in this program. In general, all alloy additions improved KIhem. The addition of 0.1% vanadium appears to be the most significant individual modification to ESR 4340 steel in that it alone provides the same gains as the more heavily alloyed ESR 4340 steels. The results must be somewhat qualified because the hardness of ESR 4340V was 50 HRC instead of the intended 53 HRC. Silicon additions of about 1.5% tended to maximize the benefits from alloy modifications. Although within an alloy system the heat treatment effects were minor or secondary relative to alloy additions, the use of an intermediate quench and subzero cooling appeared to maximize the benefits from heat treatment. Increasing the threshhold KIhem was not directly related to tempering temperature as anticipated. Overall, KIhem was increased from 10 ksi SQR(in.) to a maximum value of about 15 ksi SQR(in.). Other improvements must be based on nonconventional approaches to thermal processing.

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STUDY OF EFFECTS OF ALLOYING AND HEAT TREATMENT ON HYDROGEN EMBRITTLEMENT SUSCEPTIBILITY OF ELECTROSLAG REMELTED 4340 STEEL
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