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Dec. 57

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HIGH TEMPERATURE ACCELERATED STRESS-CORROSION OF NICKEL AND MONEL IN SULFIDE ATMOSPHERES (U)

BY

H. E. KLEIN

O.O. PROJECT: TB4-004, HIGH TEMPERATURE MATERIALS
D/A PROJECT: 5B93-32-004
REPORT NO.: WAL TN 349/58[redacted]
FILING TITLE: STRESS-CORROSION

DECEMBER 1957

[redacted]

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HIGH TEMPERATURE ACCELERATED STRESS-CORROSION OF NICKEL AND MONEL IN SULFIDE ATMOSPHERES (U)

TECHNICAL NOTE

By

H. E. KLEIN

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D/A Project: 5B93-32-004
Report No.: WAL TN 349/58[redacted]
Filing Title: Stress-Corrosion

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TITLE

HIGH TEMPERATURE ACCELERATED STRESS-CORROSION OF NICKEL AND MONEL IN SULFIDE ATMOSPHERES (U)

ABSTRACT

A study was made to determine the feasibility of utilizing wire specimens in evaluating the accelerated stress-corrosion susceptibility of nickel and Monel at elevated temperatures in sulfide environments. Hydrogen sulfide and various mixtures of hydrogen sulfide and helium were used as corrosive media. It was found that such environments caused marked reduction in time to failure for nickel and Monel. Most rapid failure occurred at high concentrations of hydrogen sulfide, but the relationship between failure time and hydrogen sulfide was not linear. The effects observed are not considered sufficiently catastrophic in nature to warrant further extensive study of gaseous sulfides in attacking agents for nickel-rich materials.

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Director
Watertown Arsenal Laboratories

REPORT APPROVED
Date 20 Dec 57
W.A.L. Board of Review
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PURPOSE AND SCOPE

The purpose of the present study was to determine the feasibility of utilizing wire specimens to investigate the possible accelerating effect of hydrogen sulfide environments in causing stress-corrosion failure of nickel and Monel at elevated temperatures. This general problem relates to a request made several years ago by Office, Chief of Ordnance to investigate the effect of harmful elements on high-nickel alloys in air-heating engines. Stress-corrosion phenomena having military implications have been under study elsewhere utilizing other metal systems under ambient conditions.

The present limited experiments were of a semi-quantitative nature, and conditions were chosen with the objective of producing rapid, decisive specimen failures. The experiments might be considered as accelerated stress-rupture tests in corrosive environments; and no attempts were made to avoid creep effects.

TEST PROCEDURE

The test materials used were in the form of wire. They were of two different compositions: *nickel*, 99.9% pure, whose spectrographic analysis showed only traces of impurities (iron, cobalt, manganese, and titanium); and *Monel* which had the following composition; 67.07% nickel, 29.53% copper, 1.29% iron, 0.94% manganese, 0.10% carbon, 0.07% silicon, and 0.011% sulfur.

The wire specimens were 0.020" in diameter and generally 27.6" in length.

The short time (30-minute) tensile properties of nickel and Monel, according to data of the International Nickel Company, are as follows:

Tensile Strength (psi)

	Room Temperature	1400°F	1500°F	1600°F
Nickel	50,000-80,000	27,000	25,000	16,000
Monel	70,000-85,000	22,000	16,000	13,000

The above data were typical values but did not represent the actual properties of the materials used. They were used only as a guide in selecting test loads.

Hydrogen sulfide was employed as the corrosive agent and helium as the inert gas and diluent. Both gases had a nominal purity of 99.9%.

The test apparatus, shown in Figure 1, consisted, essentially, of a testing furnace and a gas mixing and flow system. A separate furnace was used to pre-heat the gas before it entered the test furnace.

The pre-heater furnace in which the gas temperature was raised to 1600°F was regulated by a Leeds & Northrup Micromax Controller using a Chromel-Alumel thermocouple. The glass tubing running through the furnace was made of Vycor.

The Nichrome-wound testing furnace was set-up vertically on a ring stand. A ceramic tube was inserted in the furnace and secured in position by means of asbestos-covered clamps on a second ring stand. Transite plugs were inserted in the ends of the tube, the top plug having three drilled holes for the thermocouple, gas outlet tube, and wire specimen, respectively; and the bottom plug having two holes, one for the gas inlet tube, the other serving as a port for the specimen. The wire was supported at both ends by copper rods. It was inserted into longitudinal holes drilled in rod ends and was secured by set screws. The fixed upper rod was clamped to a ring stand. The load was applied by attaching a weight to the lower rod (Figure 2).

Specimen wire temperature was determined by making resistance measurements of the wire using a decade box and galvanometer. A variable transformer was used to regulate the power input to the furnace to reduce temperature fluctuations. Tap water, passed through copper coils, was used to cool the ends of the tube.

Wire was selected as the form for the test specimens for several reasons; convenience in static loading (the small cross-section of the wire gives a high unit stress for a given dead-weight load); ready availability in pure form, metallographic uniformity, easy temperature control while under test, uniform corroding conditions, and ease of making reproducible tests.

Several stress loads were explored, but because interest was in short time failures the present loads were employed in preference to others.

DATA

The time to failure for nickel and Monel wire under tensile stress at elevated temperatures were plotted against the concentration of hydrogen sulfide. These are shown in Figures 3 and 4.

The results show that hydrogen sulfide has a drastically corrosive effect on hot, stressed nickel and Monel. For example, at 1450°F, a sample of nickel wire failed under tensile stress of 6400 psi in 1200 seconds when surrounded by an atmosphere of static air. Above a concentration of 90% hydrogen sulfide, failure occurred in 45 seconds or less under similar conditions.

Monel wire heated to 1450°F and stressed to 15,800 psi failed in air in 135 seconds and in hydrogen sulfide in 38 seconds.

Unavoidable changes in experimental conditions such as variations in the rate of exposure of the wire to the corrosive environment, and slight fluctuations in the electric heating system led to some scattering of the data. In individual cases, this caused inconsistencies, but the trends are marked and obvious.

Metallographic examination of nickel specimens showed that failures were predominantly intergranular, both in air and in hydrogen sulfide, although in the latter environment there appeared to be some evidence of transgranular failure as well (see Figures 6 and 7). The initial condition of the wire is shown in Figure 5.

Stress-corrosion failures of nickel and nickel alloys in hydrogen sulfide at temperatures exceeding 1200°F may be explained by a mechanism described by Copson[1]. He suggests that at elevated temperatures, a molten corrosion product of nickel/nickel-sulfide eutectic penetrates nickel grain boundaries, localizes tensile stresses, and accelerates attack resulting in cracking and failure.

CONCLUDING REMARKS

The present study has demonstrated the feasibility of utilizing wire specimens for investigations of accelerated intergranular failure of nickel and Monel when exposed to a hydrogen sulfide environment at elevated temperatures. The shortest failure times occurred when high concentrations of hydrogen sulfide were used, but the relationship between failure time and sulfide concentration was not linear.

Under the conditions of test, the effects produced by hydrogen sulfide were not truly catastrophic to justify larger scale experiments involving gaseous sulfides as attacking agents.

Subsequent to the work presented here, experiments have been performed with Nichrome V wire, also at elevated temperatures. The results of these tests will be summarized in a later report. Other experimental work involving ambient temperature stress-corrosion reactions of Nichrome V, steel, titanium, and other metals in contact with solid and liquid corrosive agents is also in progress.

[1] *Copson, H. R., "Laboratory Techniques for the Investigation of Stress-Corrosion Cracking," pp. 189-90 in William D. Robertson, ed., Stress-Corrosion Cracking and Embrittlement, Wiley (New York), 1956.*

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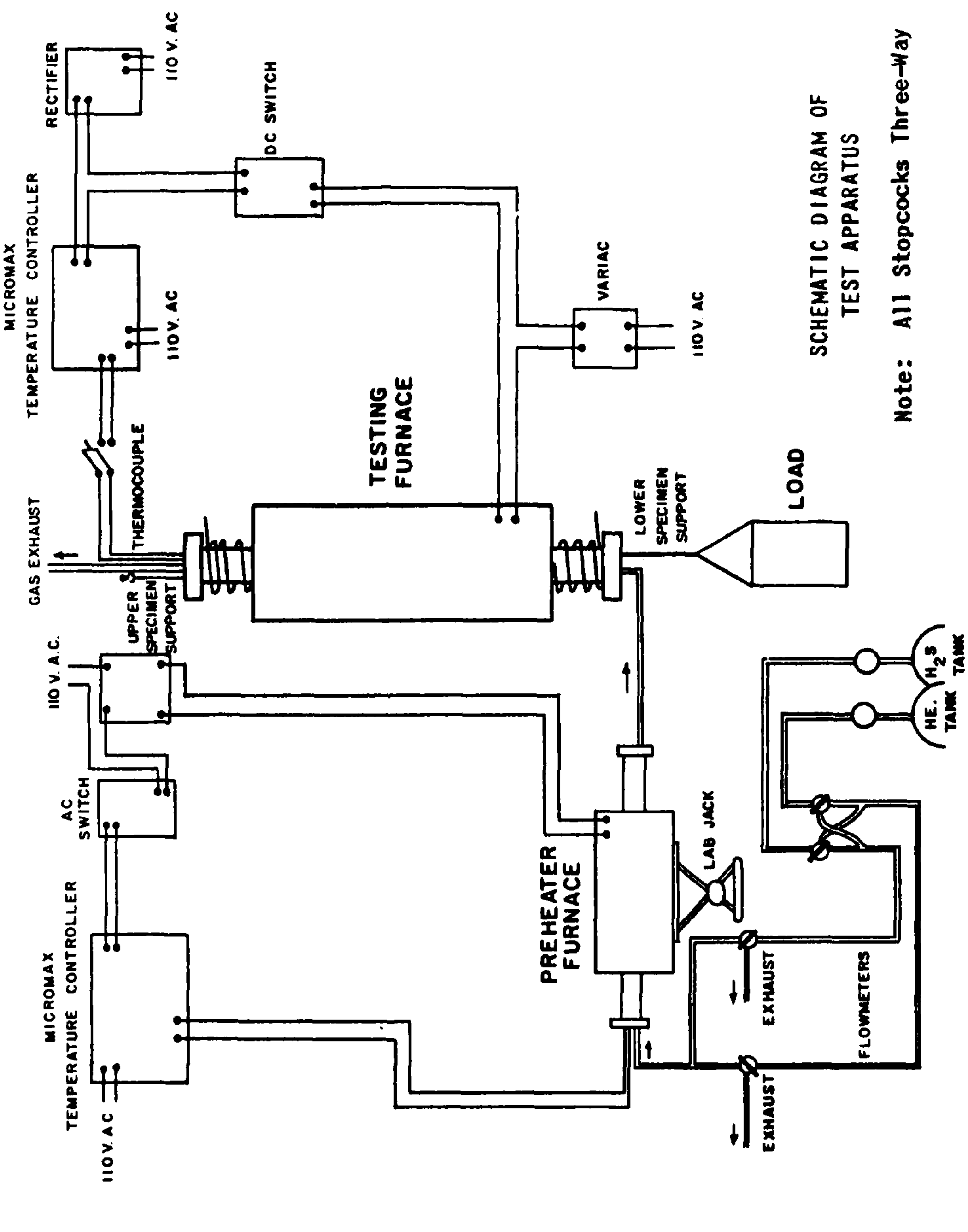
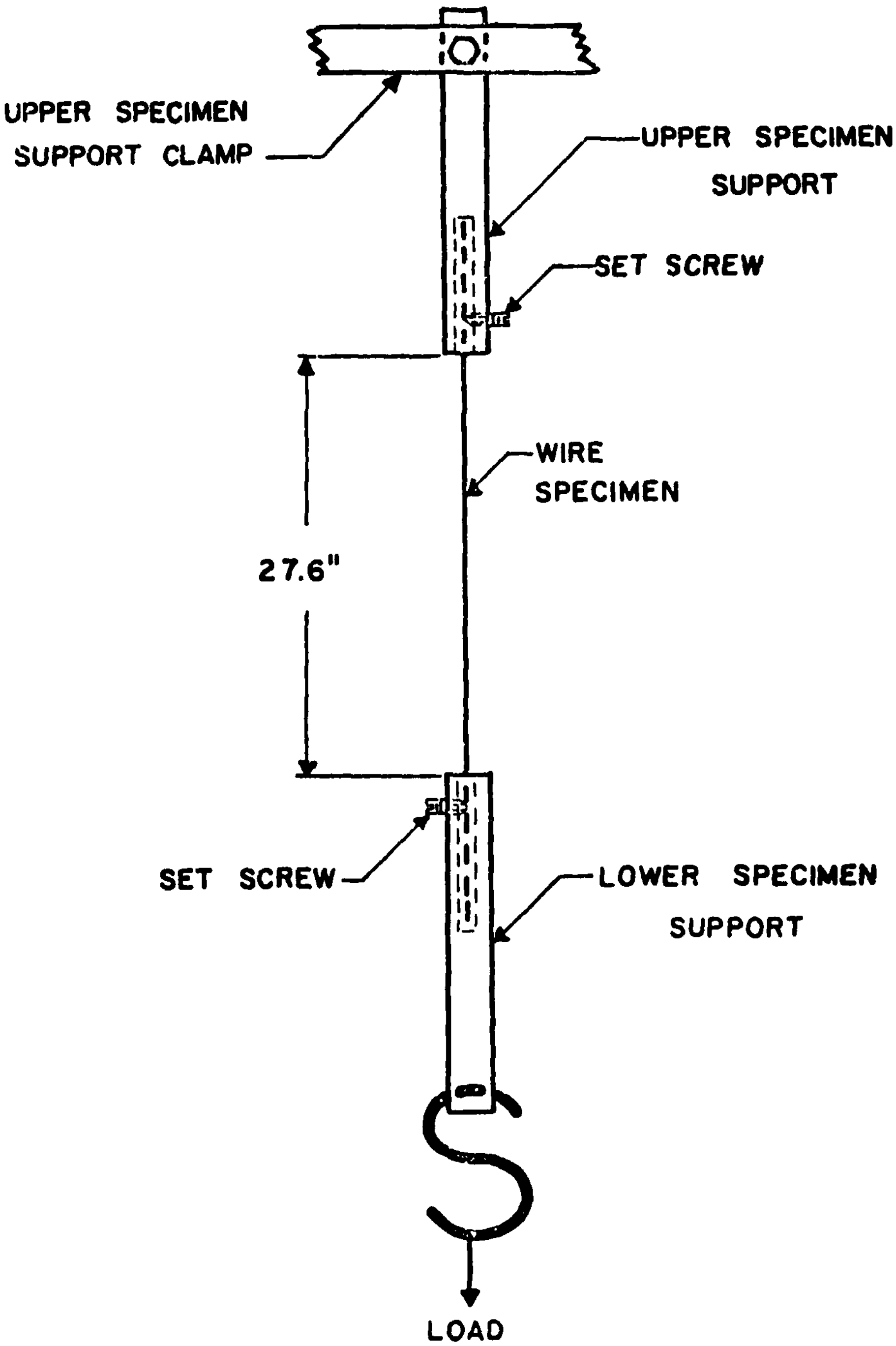


SCHEMATIC DIAGRAM OF
TEST APPARATUS

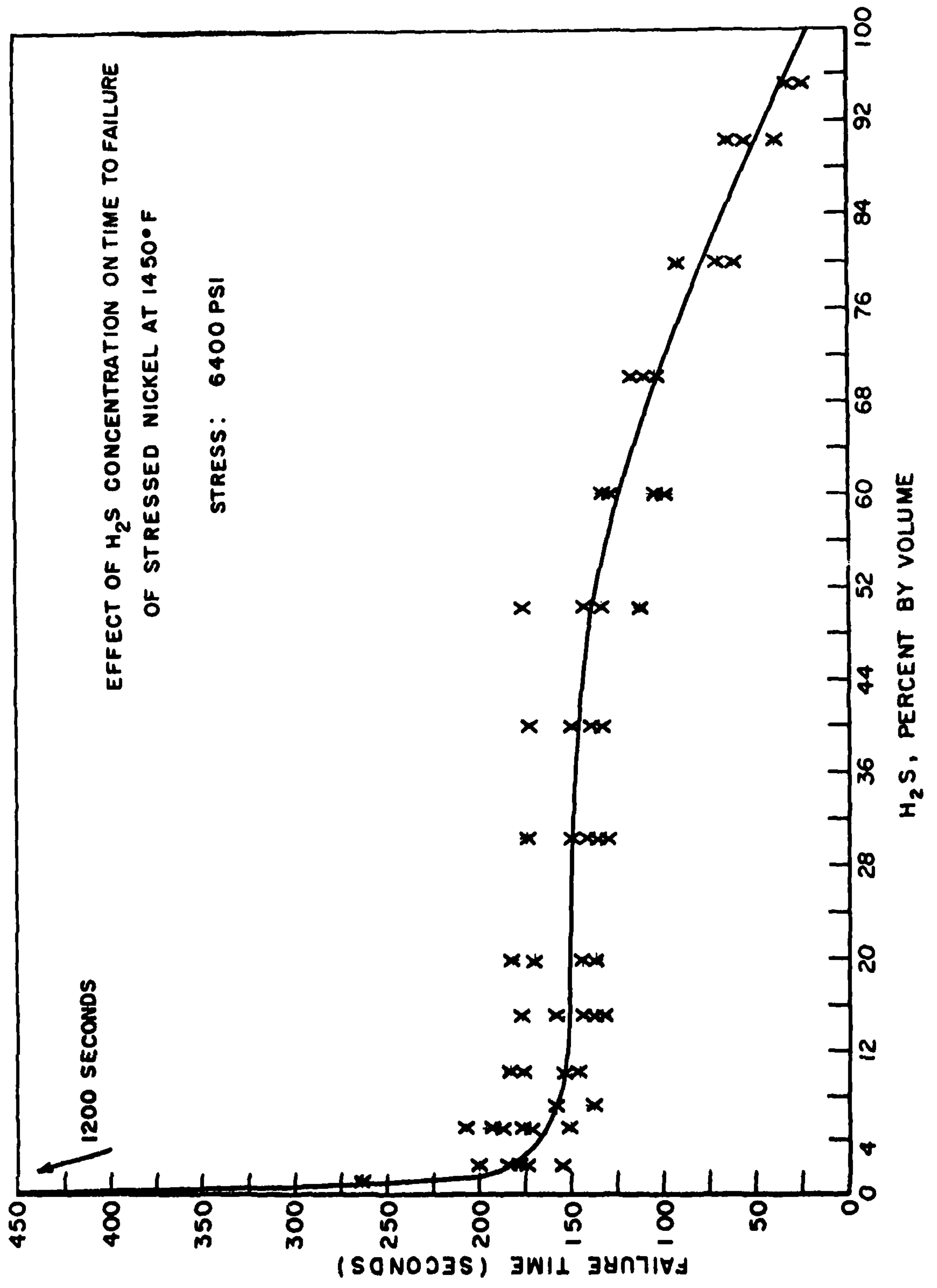
Note: All Stopcocks Three-Way

FIGURE 1



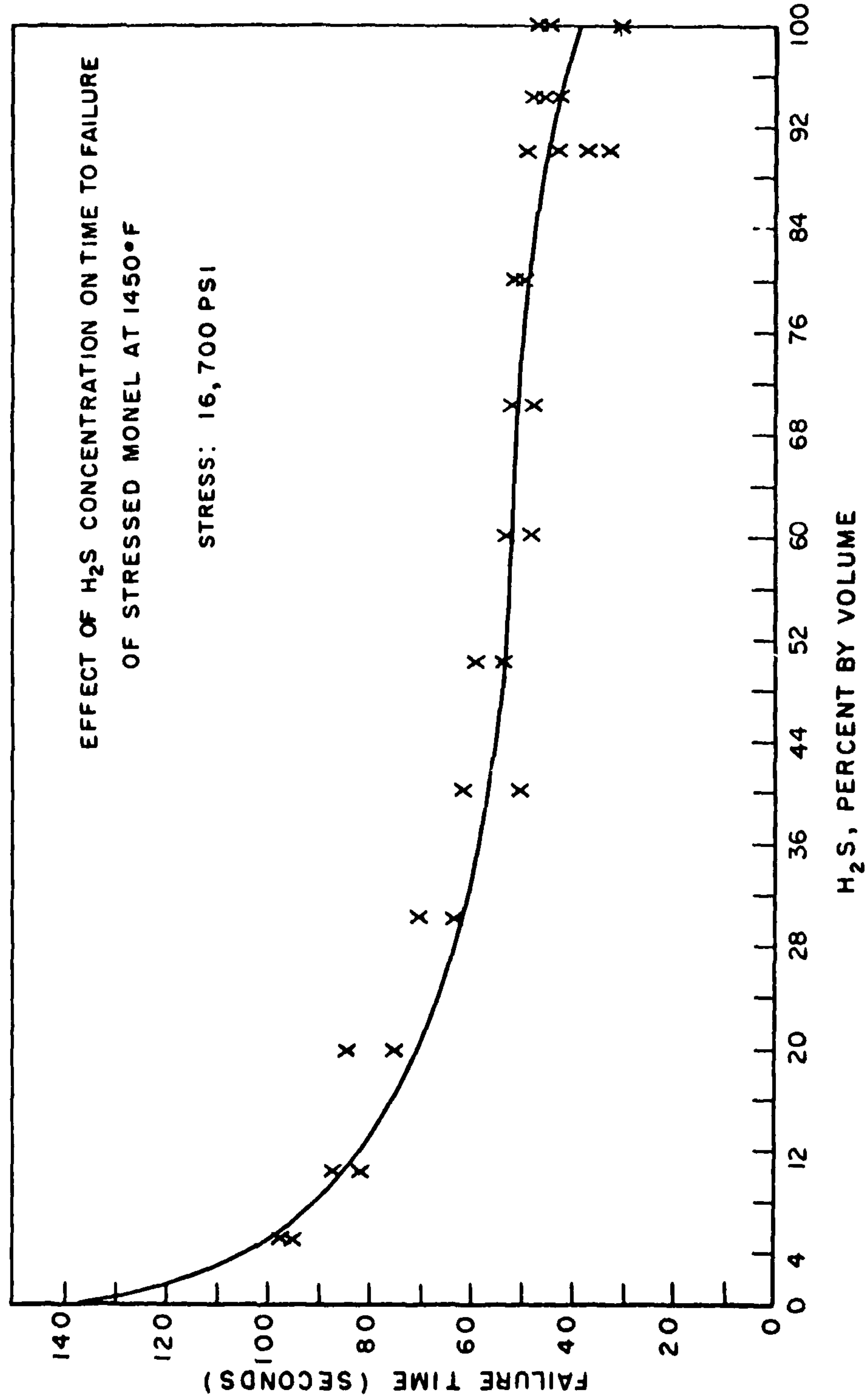
DETAIL OF SPECIMEN
CLAMPING METHODS

FIGURE 2



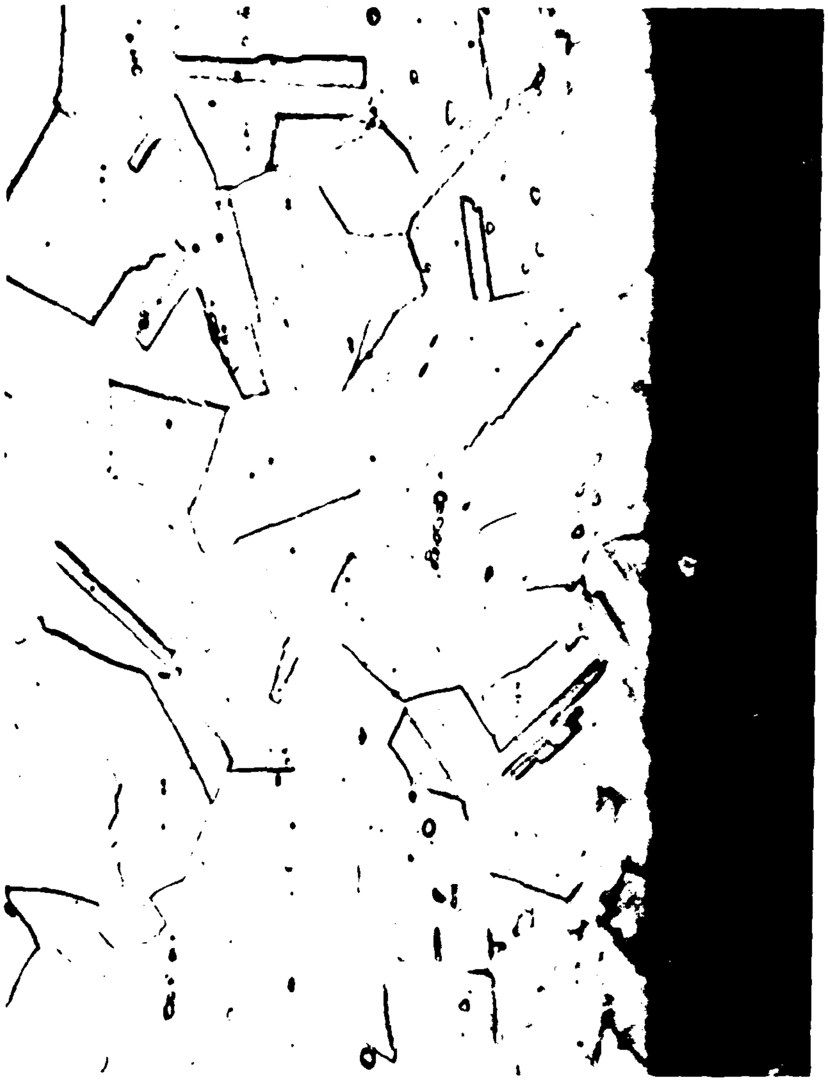
EFFECT OF H_2S CONCENTRATION ON TIME TO FAILURE
OF STRESSED NICKEL AT 1450°F
STRESS: 6400 PSI
1200 SECONDS FAILURE
FAILURE TIME (SECONDS)
0
50
100
150
200
250
300
350
400
450
H_2S, PERCENT BY VOLUME
0
4
12
20
28
36
44
52
60
68
76
84
92
100

FIGURE 3



EFFECT OF H_2S CONCENTRATION ON TIME TO FAILURE
OF STRESSED MONEL AT 1450°F
STRESS: 16,700 PSI
FAILURE TIME (SECONDS)
140
120
100
80
60
40
20
0
0
4
12
20
28
36
44
52
60
68
76
84
92
100
H_2S, PERCENT BY VOLUME

FIGURE 4



X1000 1725 17135

SECTION OF ANNEALED NICKEL WIRE AS RECEIVED — Etched in a 50/50 (by vol.) mixture of concentrated nitric and glacial acetic acids

Wtn. 639-15,972

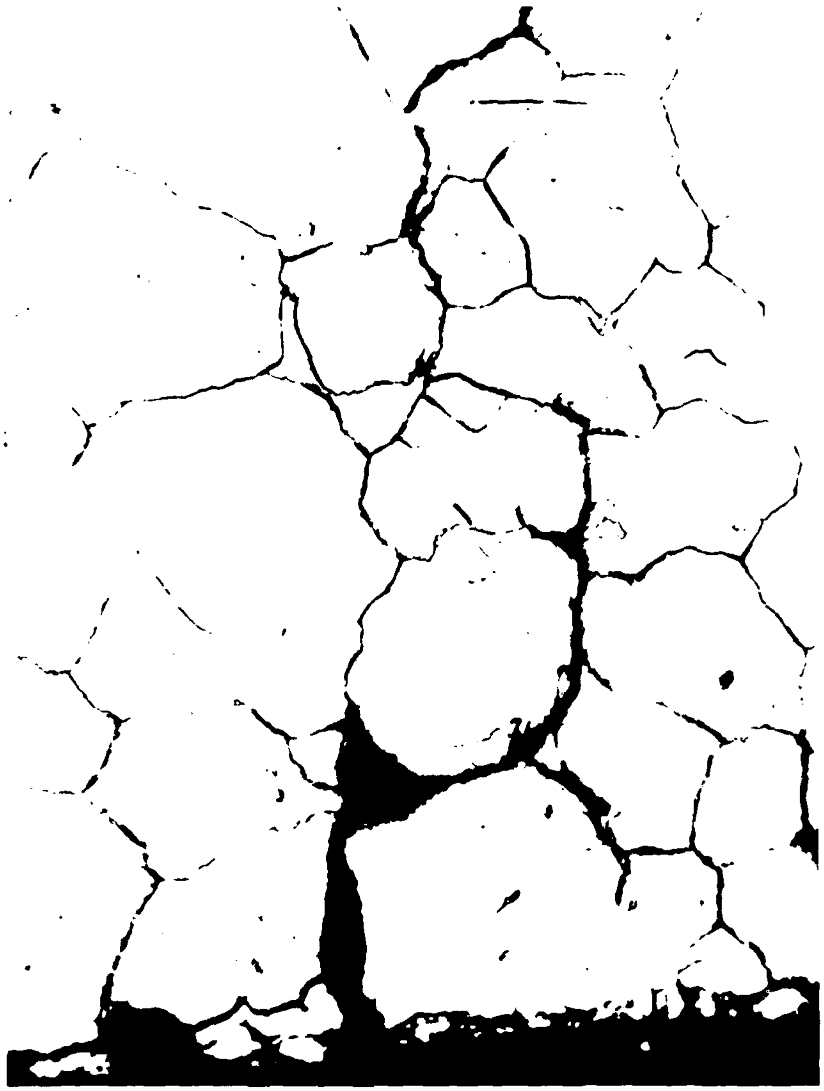
FIGURE 5

X1000 32 16502

SECTION OF NICKEL WIRE WHICH FAILED IN HYDROGEN SULFIDE UNDER A STRESS OF 6400 PSI AT 1450°F - Etched in a 50/50 (by vol.) mixture of concentrated nitric and glacial acetic acids

Wtn. 639-15,973

FIGURE 6



X1000 30 16500

SECTION OF NICKEL WHICH FAILED IN A HYDROGEN SULFIDE ATMOSPHERE UNDER A STRESS OF 6400 PSI AT 1450°F - Etched in a 50/50 (by vol.) mixture of concentrated nitric and glacial acetic acids

Wtn. 639-15,971

FIGURE 7

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