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FAILURE-ANALYSIS REPORT

DRILL PIN ✓

ENTE NAZIONALE

PER L'ENERGIA ELETTRICA (ENEL)

GRUPPO MINERARIO

LARDERELLO, ITALY

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Introduction

A cracked drill pin was received from ENEL's Sasso 22 geothermal well with a request for failure analysis. Failure reportedly occurred during temperature measurements inside the drill pipe. This drill pipe had been put into the 4000 meter (13,124 ft.) hole for instrument protection about 80 hours before temperature measurements. The temperature measured at the bottom of the hole was 380°C (716°F). The liquid level was at 2000 meters (6562 feet) depth, and the temperature at that depth was 95°C (203°F). Information on the depth where failure of this pin occurred was not given. Numerous drill string failures of this type have been reported at depths greater than 3000 meters (9843 feet) by ENEL.¹

A mixture of 70% river water and 30% condensate from a nearby geothermal power plant was being used as drilling fluid. The average chemical composition of the power plant wastewater is given in Table 1. This region of Italy is known as the Boraciferous Region. The average chemistry of the Boraciferous geothermal fluid is given in Table 2. The failed drill pin was reportedly made of 38 NC D4 steel.

Examination Procedure

The cracked drill pin is shown in Figure 1 as-received. Figure 2 shows the inside surface with the crack penetrating through the wall. Internal deposits were removed for energy dispersive x-ray (EDX) analysis to determine the elements present, and for x-ray diffraction (XRD) to identify compounds. There were no deposits on the outside of the drill pin. The pin was then sectioned transversely through the threaded portion, and cut off at the last thread for metallographic and fractographic examination. The crack opened during sectioning, and the threaded portion formed three pieces as shown in Figure 3. Hardness readings were taken transversely on the cut edge of piece 4 in Figure 3. A portion of piece 3 was chemically analyzed to confirm the material composition of the drill pin. A section of piece 4 was cut through the crack and prepared for metallography by standard metallographic techniques. A portion of the fracture face of piece 2 was cut for fractographic examination in the scanning electron microscope. This piece was electrolytically cleaned with an electroplater's cleaning solution to remove the scale for viewing the fracture surface.

Examination Results

The EDX analysis will detect elements with atomic numbers greater than 11 (sodium). The elements detected by EDX on scale from inside the drill pin were iron, sulfur, calcium, silicon, and chromium. The compounds identified in this scale by XRD were iron chromium oxide (Cr_2FeO_4), chromium oxide (CrO), iron oxide (Fe_2O_3), calcium iron silicate ($\text{Ca}_3\text{Fe}_2(\text{SiO}_4)_3$), and iron sulfide (FeS).

The results of the chemical analysis of the drill pin metal are shown in Table 3 with the specification for 38 NC D4 steel and its U.S. equivalent, American Iron and Steel Institute (AISI) 9840. The chemical analysis of the drill pin confirmed that it was made of 38 NC D4 as reported.

The hardness readings along the transverse section ranged from Rockwell C 33 to Rockwell C 36 with an average of Rockwell C 35.2. This corresponds to an approximate tensile strength of 1089 MPa (158,000 psi).² There was no significant hardness gradient on the area analyzed.

The section prepared for metallography showed a martensitic structure as seen in Figure 4. This needle-like pattern is typical of a high strength low alloy heat treated steel. The inside surface of the pin showed some pitting when viewed in the metallographic cross-section. Microcracks emanated from some of these pits.

The fracture surface analyzed by scanning electron microscopy is shown in Figure 5. Most of the fracture surface displayed a distinct intergranular mode of failure as shown in Figure 6. This fracture was initiated on the inside surface where the failure mode was entirely intergranular. About 60 percent of the way through the wall the intergranular fracture transformed to microvoid coalescence (dimple formation). Figure 7 is a scanning electron micrograph of this transition area of the fracture surface. Near the outside surface the failure mode is microvoid coalescence with some tearing at final fracture. This is shown in the dual magnification fractograph of Figure 8.

Conclusions

This drill pin failed by wet H_2S cracking. Such high strength steel in the hydrogen sulfide environment of this geothermal well is most susceptible to this type of failure. The resultant brittle failure is well known in the oil industry as sulfide stress cracking, sulfide corrosion cracking, or hydrogen embrittlement.³

Discussion

Most of the compounds identified by XRD were the result of oxidation after the failure. The iron sulfide came from the corrosion reaction of steel with aqueous H_2S in the geothermal fluid and/or power plant condensate. This reaction is also the source of the hydrogen which entered the steel and caused the brittle failure.^{3,4} Increasing concentrations of hydrogen sulfide shorten the time to failure, but failures may still develop at concentrations of only a few parts per million.⁴ In this case, the several hundred parts per million of H_2S in the geothermal fluid would be more than adequate to induce cracking.

Sulfide stress cracking can progress by intergranular cracking, microvoid coalescence, and cleavage modes. The fracture mode depends on the stress, concentration of hydrogen at the crack tip (i.e., H_2S concentration), microstructure, and whether hydrogen is present in the material before the crack tip is stressed or is admitted into the crack after stressing. Intergranular cracking and cleavage are expected where the metal is charged with hydrogen prior to crack formation. Intergranular cracking usually occurs at low applied stresses, generally below 345 MPa (50,000 psi). Microvoid coalescence is expected where hydrogen is added at the growing crack tip, and at higher applied stress, greater than 483 MPa (70,000 psi).⁵ In this drill pin,

a combination of intergranular cracking followed by microvoid coalescence was observed. This indicates that the metal was charged with hydrogen for a period before cracking was initiated and that hydrogen continued to be added during crack growth. Cracking initiated at the pits as observed on the inside surface. Crack growth through the wall of the drill pin caused a reduction in cross sectional area and an increase in applied stresses. When the applied stresses reached a threshold of about 483 MPa (70,000 psi), the mode of failure changed from intergranular to microvoid coalescence as observed on the fracture surface. The tearing and final fracture probably followed relatively quickly.

Another important consideration is the strength and hardness of the metal exposed to hydrogen sulfide. The minimum amount of hydrogen sulfide required for failure decreases as the strength and hardness of the steel are increased. The National Association of Corrosion Engineers has attempted to define steels that are resistant to cracking as those with a hardness below Rockwell C 22.⁶ Hardness was selected because it was a convenient and nondestructive measurement of strength. The drill pin average hardness of Rockwell C 35 is well above the threshold limit for sulfide stress cracking. However, experience has indicated that a hardness limit is a simplified approach. All the factors which determine the metallurgical properties of a steel have an interdependent influence on its resistance. Steels must be considered in terms of composition, heat treatment, mechanical working, and the resultant microstructure. It is generally agreed that untempered martensite has a marked adverse effect on resistance to cracking in sulfide environments. Steel compositions and heat treatments that give uniform structures are desirable. Quenched and tempered steels are considered to be more resistant to cracking than normalized and tempered steels, which in turn are more resistant than normalized steels.³ Cold work of the steel prior

to exposure increases residual stress, thereby reducing the resistance to sulfide cracking.

Other environmental factors to consider in sulfide stress cracking are pH and temperature. Lowering the pH significantly enhances cracking. Cracking has been shown to be greatly decreased at pH above 6 and absent at pH 9.5.⁷ Increasing temperature generally retards cracking by homogenizing the hydrogen and thereby preventing local concentrations.⁴ The temperature at the bottom of this hole was 380°C (716°F) and would generally be high enough to offer some resistance to cracking. However, it was not known how close to the bottom this drill pin failure occurred. Sulfide stress cracking could have occurred when the drill pin was removed from the higher temperature area, and this drill pin could have been previously exposed to H₂S in lower temperature wells.

Recommendations

In most aqueous sulfide drilling situations, strength requirements and economic limitations restrict the materials selection to low alloy steels. In these environments, the minimum strength drill pipe with a hardness of less than Rockwell C 22 should be selected, if possible. This may not be possible for strength requirements needed in such deep wells. Then as a minimum, quenched and tempered heat treatment should be specified to give uniform microstructures that will provide improved resistance to sulfide cracking. Steels should also be heat treated after cold working, and precautions should be taken to minimize cold working during use. Some drilling fluids are used which have some protective properties and minimize the contact of H₂S with the drill string. Alkaline drilling fluids are used in this way to neutralize minor hydrogen sulfide contamination.

References

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TABLE 1. CHEMICAL COMPOSITION OF GEOTHERMAL POWER PLANT
CONDENSATE USED FOR DRILLING FLUID.⁸

pH = 7.4

H ₂ S	0.23	cm ³ /l
H ₃ BO ₃	635	ppm
NH ₄ ⁺	132	ppm
Cl ⁻	1.6	ppm
K ⁺	0.08	ppm
Na ⁺	2.2	ppm
Ca ⁺⁺	4.0	ppm
Mg ⁺⁺	0.2	ppm
Fe ⁺⁺	0.6	ppm
SO ₄ ⁼	147.2	ppm
SiO ₂	4.9	ppm

TABLE 2. AVERAGE CHEMISTRY OF GEOTHERMAL FLUID IN
THE BORACIFEROUS REGION OF ITALY.⁹

H ₂ O	951,010	ppm
CO ₂	47,550	ppm
H ₂ S	540	ppm
H ₃ BO ₃	220	ppm
NH ₃	150	ppm

TABLE 3. CHEMICAL COMPOSITION OF DRILL PIN
(weight percent)

<u>Element</u>	<u>Chemical Analysis</u>	<u>38 NC D4¹⁰</u>	<u>AISI 9840¹¹</u>
C	0.39 ± 0.01	0.34 - 0.42	0.38 - 0.43
Si	0.21 ± 0.006	0.40 max.	0.20 - 0.35
Mn	0.75 ± 0.02	0.50 - 0.80	0.70 - 0.90
P	not analyzed	0.035 max.	0.040 max.
S	0.013 ± 0.0004	0.035 max.	0.040 max.
Ni	0.84 ± 0.025	0.70 - 1.0	0.85 - 1.15
Cr	0.85 ± 0.025	0.70 - 1.0	0.70 - 0.90
Mo	0.13 ± 0.004	0.15 - 0.25	0.20 - 0.30

Figure 1. Cracked drill pin as-received.

Figure 2. View of the inside surface showing crack extending through the wall.

Figure 3. Pieces numbered for identification after sectioning. Pieces 2 and 3 are the fracture faces which fell apart during sectioning.

Magnification: 800X
Etchant: 2% Nital

Figure 4. Typical martensitic microstructure of the drill pin.

Magnification: $\approx 4X$

Figure 5. Macrograph of the fracture face after cleaning. The crack initiated on the inside surface which is at the bottom of the photo.

Magnification: 250X

Figure 6. Area of intergranular cracking taken from near the bottom center of Figure 5.

Magnification: 250X

Figure 7. Transition area where fracture mode changed from intergranular at the bottom to microvoid coalescence at top center. Taken from the upper middle of Figure 5.

Magnification: Left - 250X
Right - 1000X

Figure 8. Area of microvoid coalescence and final fracture tearing from the top middle of Figure 5. The fractograph on the right is a higher magnification view of the outlined region on the left. It displays a dark void and light-toned tear ridges.