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**Analysis of Level Control Valve Seat
from the Recycle Condensate Separator
at the Ft. Lewis, Washington,
SRC Pilot Plant**

V. B. Baylor

OAK RIDGE NATIONAL LABORATORY
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METALS AND CERAMICS DIVISION

**ANALYSIS OF LEVEL CONTROL VALVE SEAT FROM THE RECYCLE CONDENSATE
SEPARATOR AT THE FT. LEWIS, WASHINGTON, SRC PILOT PLANT**

V. B. Baylor

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ANALYSIS OF LEVEL CONTROL VALVE SEAT FROM THE RECYCLE CONDENSATE SEPARATOR AT THE FT. LEWIS, WASHINGTON, SRC PILOT PLANT

V. B. Baylor

ABSTRACT

A seat from the level control valve for the recycle condensate separator at the Ft. Lewis Solvent Refined Coal pilot plant was removed from service when cracks were noticed during routine maintenance. One crack extended completely through the 17-4 PH stainless steel trim set. Metallographic examination and hardness measurements revealed that the aging temperature used in heat-treating the martensitic precipitation-hardenable steel was too low, resulting in a material with an improper balance of tensile and impact properties. Residual stresses due to fabrication probably combined with the corrosive environment to produce stress corrosion cracking. The offensive species could not be positively identified, but hydrogen is thought to be a contributory factor.

INTRODUCTION

Background and Source of Part

The Solvent Refined Coal (SRC) process affords a promising method for converting high-sulfur coals to low-sulfur fuels through coal liquefaction. Coal is liquefied by slurring pulverized coal with process-derived solvent, and then mixing with hydrogen at 427 to 454°C (800–850°F) and 11.7 MPa (1700 psi) to dissolve most of the coal. Sulfur is converted to hydrogen sulfide, which is removed as a gas. Various separation techniques yield different fuels which are environmentally safer than the original coal.

There are two different modes of operation, called SRC-I and SRC-II. The processes differ in terms of the amount of hydrogen that can be injected into the coal molecules in the reactor, the type of product separation, and the nature of the product. The SRC-II mode effects an increased ratio of hydrogen to carbon in the slurried coal, producing a more refined liquid with a lower boiling point. Distillation gives a low-sulfur fuel oil. The SRC-I mode produces a less-refined liquid, which cools to form a low-sulfur solid fuel, which is nearly ash-free.

A 50 ton/d SRC Pilot Plant is being operated by the Pittsburgh and Midway Coal Mining Company (P&M), a subsidiary of Gulf Oil Corporation, in Ft. Lewis, Washington. Sponsored by the Department of Energy, the plant was designed by P&M and built by Rust Engineering in 1974. It can be operated in either the SRC-I or SRC-II mode. Further details on the operating history are available.¹

Description of Part

The valve seat is shown in Fig. 1 as it was received at the Oak Ridge National Laboratory. The valve seat is part of a standard high-pressure letdown valve manufactured by Fisher Valve Company but contained no identifying marks. The trim was stated to be 17-4 PH stainless steel, which is commonly used for valve trim.

¹Pittsburgh and Midway Coal Mining Company, *Development of a Process for Producing an Ashless, Low-Sulfur Fuel from Coal: Volume III – Pilot Plant Development Work, Part 3 – Startup and Operation of the SRC Pilot Plant*, FE-496-T13 (May 1977).

Y-155952

(a)

Y-155951

(b)

Fig. 1. Valve Seat from the Level Control Valve for the Recycle Condensate Separator at the Ft. Lewis Solvent Refined Coal Plant as Received at ORNL. (a) Top of the seat showing crack causing failure. (b) Bottom of the seat showing two smaller cracks in addition to the crack seen in (a).

It was shrink fitted around the tungsten carbide insert. The 23.2-mm-long insert has a 19.1 mm OD and a wall thickness that varies, because of the tapered orifice, from 11.0 to 12.7 mm, with a wall thickness of 12.5 mm in the middle. The trim is 24.7 mm long, including the raised portions on each end. It has a 47.7 mm OD with a wall thickness of 14.2 mm through the central portion.

Description of Service Conditions

The valve seat was part of a level control valve for the recycle condensate separator at the Ft. Lewis SRC Plant. This component separates a mixture of process gases into vapor and liquid components. The vapor stream passes through a pressure control valve to the acid gas treatment area; the liquid goes through the level control valve. This valve serves to let down nominal pressures from approximately 7 to 0.7 MPa (1000 to 100 psi) at ambient temperature, nominally 38°C (100°F). The environment is recycle water, solvent, low-boiling distillate liquids, and a small amount of H₂S and CO₂.

The valve was installed in 1976. The cracking visible in Fig. 1 was noticed during routine maintenance, and the seat was removed at an unknown date. It was received for examination at ORNL in June 1978. Since it was the only valve seat of its kind to have failed in this location, it was replaced with the same type.

ANALYSIS

The cracks in the valve seat can be clearly seen in Fig. 1. Two small cracks stopped about halfway down the side. The third crack is the major defect; it extends from top to bottom and through the entire wall thickness of the trim. All cracks lie within about 150° of each other with no pattern of symmetry.

In preparing samples for analysis, the trim set was first cut by sawing the side 180° from the major crack and then carefully cutting between the fracture surfaces to halve the piece and remove the insert. Three pie-shaped sections were cut in the cracked half; one to guarantee a fracture surface for scanning electron microscopy, one for chemical analysis, and a large section for metallographic examination of the smallest crack. Further sectioning was done to reveal the middle of the piece and a radial plane.

A small sample was chemically analyzed semiquantitatively. The results shown in Table 1 confirm the identification of the material as 17-4 PH, which is a martensitic precipitation-hardenable stainless steel.

Figure 2 is an unetched cross section of the low-pressure side of the trim, showing the smallest crack. Figure 3 shows the crack magnified in the unetched condition. Note the secondary branching in Fig. 3(a). Closer inspection (b) reveals multiple small cracks, some of which are not connected to the main crack in the polished plane, although subsurface branches probably connect the cracks.

The sample was etched with Fry's reagent to investigate the microstructure and nature of the crack path and secondary branching (Fig. 4). Generally, 17-4 PH is solution treated at 1038°C (1900°F) and cooled in oil or air below 32°C (92°F), followed by aging at 482 to 621°C (900–1150°F) and air cooling. The martensite that forms as a result of the solution treatment provides strength and hardness in the material. The aging treatment stress relieves the martensite, increasing the ductility and toughness, and allows the precipitation of a copper-rich second phase, increasing the strength^{2,3} over that achieved by solution treatment (assuming no overaging). The amount and nature of the martensitic structure are determined by

²D. C. Perry and J. C. Jasper, "Structure and Constitution of Wrought Precipitation-hardenable Stainless Steels," pp. 7-1–7-18 in *Handbook of Stainless Steels*, ed. by D. Peckner and I. M. Bernstein, McGraw-Hill, New York, 1977.

³G. E. Linnert, "Welding Precipitation-Hardening Stainless Steels," *Weld. J. (New York)* 36:9–27 (1957).

**Table 1. Chemical analysis of valve trim
compared with ASTM chemical
specifications for 17-4 PH**

Element	Content, wt %	
	Valve trim	Type 630 ^a (17-4 PH)
C		< 0.07
Mn	0.3	< 1.00
P		< 0.04
S		< 0.03
Si	0.5	< 1.00
Cr	16.3	15.50–17.50
Ni	4.5	3.00–5.00
Cu	3.3	3.00 5.00
Nb + Ta	0.30	0.15–0.45

^aASTM Specification A 564-74, "Standard Specification for Hot-Rolled and Cold-Finished Age-Hardening Stainless and Heat-Resisting Steel Bars and Shapes," *1977 Annual Book of ASTM Standards*, American Society for Testing and Materials, Philadelphia, 1977, pp. 463–69.

**Fig. 2. Smallest Crack Across Bottom of Polished and Unetched Trim Set. Some secondary branching can be seen.
10X.**

Fig. 3. Smallest Crack Across Bottom Surface of Valve Trim. Note that the secondary branching remains close to the main crack, extending no further than 0.13 mm (0.005 in.) into the metal. As polished (a) 100X, (b) 500X.

the aging temperature. The microstructure shown in Fig. 4 is typical of the tempered martensitic transformation produced by the H925 condition,⁴ which can be achieved by aging at 495°C for 4 h followed by air cooling.

Hardness readings were taken to verify the heat treatment as H925 by use of a Kentron tester with a 1000-g load and a 20X objective. Microhardness values were determined across the top of the mounted sample away from the crack and also longitudinally along a plane normal to the top. Hardness readings are listed in Table 2. The hardness ranged between 40 and 44 Rockwell C, confirming that the maximum aging temperature was around 495°C or below.

Since the transformation to martensite was essentially complete, the prior austenite grain boundaries were not easily delineated by etching. A few grain boundaries can possibly be distinguished, but the cracking cannot really be described as solely transgranular or intergranular. Cracks appeared both through and around grains but were predominantly transgranular. In some instances, the small secondary cracks seem to parallel the orientation of the acicular martensite structure [Fig. 4(b)].

In general, as the aging temperature decreases below 621°C, the strength and hardness increase while the ductility and impact strength decrease. In the H925 condition the acceptable room-temperature Charpy V-Notch impact strength is no lower than 8 J (5 ft-lb), and for H900 (age at 482°C) minimum impact properties are not even specified.⁵ The heat treatment of the trim thus yielded a very strong, but very brittle, material.

Residual stresses were undoubtedly present in the material as a result of fabrication. They can originate from heat shrinking as a result of dissimilar-metal contraction upon cooling. Stresses may also originate from poor fit, as shown in Fig. 5, or from the hardening treatment, specifically quenching. Concentration of stresses at sharp corners, the reduced impact strength, and reduced notch ductility combine to increase the probability of brittle cracking.

The brittleness of the failure is shown in the micrographs. Secondary branching is not extensive as is typical of predominantly brittle failures in hardenable stainless steels.⁶ In addition, no evidence of typical internal stress relief (other than cracking) or plastic deformation can be seen.

The fracture surface from the major crack (Fig. 6) shows cleavage with no obvious regions of plastic deformation. The surface was relatively clean, with no oxide buildup. The arrow points to an outside corner where the crack may have initiated. This area is more level than any other region of the fracture surface. Some evidence of striations was seen, but they were not well-developed or extensive. The surface did not have the shiny appearance of a classical brittle fracture, possibly because some corrosion products were present, yet it displayed none of the classical aspects of a ductile or shearing mode of failure, such as cups or cones. Toward the inner surface of the trim the fracture surface appeared somewhat more jagged. The crack may have initiated along the outside surface of the trim and then propagated by cleavage through the wall thickness.

The question remains whether the material could have cracked solely due to residual stresses present from fabrication or if the environment was a factor, implying stress corrosion cracking or hydrogen embrittlement. The rough grainy appearance of the outside edges of the fracture surface in Fig. 6 has been noted previously in cases of stress corrosion cracking of hardenable stainless steels.⁶ Type 17-4 PH stainless steel can be sensitized by aging at too low a temperature, and fresh water has been observed to cause stress corrosion cracking in 17-4 PH at room temperature.⁷ The presence of H₂S, which often causes stress

⁴ *Metals Handbook*, 8th ed., Vol. 7 – *Atlas of Microstructures of Industrial Alloys*, American Society for Metals, Metals Park, Ohio, 1972.

⁵ *Armco Precipitation-Hardening Stainless Steels*, Armco Steel Corp., Middletown, Ohio, October 1977.

⁶ F. K. Bloom, "Stress Corrosion Cracking of Hardenable Stainless Steels," *Corrosion*, 11(8):351t–361t (August 1955).

⁷ *Metals Handbook*, 8th ed., Vol. 10 – *Failure Analysis and Prevention*, American Society for Metals, Metals Park, Ohio, 1975.

Fig. 4. Same Crack Location Shown in Fig. 3, Etched in Fry's Reagent. Note structure characteristic of 17-4 PH in H925 condition with almost complete transformation to martensite. Also note the random nature of the cracking network. (a) 100 $\times$, (b) 500 $\times$.

Table 2. Hardness readings from top plane and normal radial face of 17-4 PH stainless steel trim set

Hardness of Top ^a		Hardness of Face ^b	
(DPH)	(Rockwell C)	(DPH)	(Rockwell C)
452.7	44	418.6	40
431.8	42	412.6	40
418.6	40	425.8	41
425.1	41	438.6	43
425.1	41	431.7	42
		445.6	44
		438.6	43
		438.6	43

^aReadings are from inner to outer surface along a radius.

^bReadings are from top to bottom.

Fig. 5. Polished Surface of Radial Plane of Trim Set. The inside of the piece is on the left. Note the smoothness of the inner surface until just above the bottom of the photograph. The surface is jagged and indented, a condition that can be caused by a poor fit. 10X.

Fig. 6. Fracture Surface of Major Crack. Little evidence is seen of ductile failure on the surface. The crack may have initiated in an outside corner (arrow). Shiny areas are due to saw cut during sectioning. 4X.

corrosion cracking, further supports the probability that the cracks resulted from residual stresses combined with the corrosive effects of the environment. The cracking could be due to hydrogen attack. This phenomenon has been observed in 17-4 PH in the H900 condition at room temperature⁷ but is generally hard to distinguish from conventional stress corrosion cracking in precipitation-hardenable stainless steels. The grainy texture of the fracture surface noted above and the primarily transgranular nature of the cracking with little secondary branching have been seen⁶ in cases of stress corrosion cracking where hydrogen was thought to be a contributory factor. In fact, a proposed⁶ mechanism of stress corrosion cracking in hardenable stainless steels involves the absorption of hydrogen generated by corrosion, which accelerates crack propagation.

Scanning electron microscopy supported the conclusion that the predominantly brittle cleavage resulted from residual stresses in combination with the environment. Fig. 7(a) shows the grainy texture of the outside edge of the fracture surface. Successively closer investigation [Fig. 7(b) and (c)] shows that the fracture is primarily brittle in this area. Figure 8 shows an area towards the inner surface of the trim, where the surface is not as smooth (a). Further magnification reveals areas that resemble classical regions of hydrogen embrittlement in the valleys (arrow). The micrographs also confirm the lack of oxide buildup noted above.

Fig. 7. Scanning Electron Micrographs of Outer Edge on Fracture Surface of Major Crack. (a) 100X (b) 300X (c) 1000X. Note grainy texture shown in (a) and brittle cleavage shown in (c).

Fig. 8. Scanning Electron Micrographs of Interior Region of Fracture Surface. Here the cleavage does not appear as brittle as it does in Fig. 7. (a) 100X (b) 300X (c) 1000X. Ledges in the valley in (c) (arrow) may indicate subsurface activity due to absorption of hydrogen.

CONCLUSIONS

From the observations and operating history of the level control valve seat that cracked in the Ft. Lewis SRC Pilot Plant, these conclusions were reached:

1. The 17-4 PH stainless steel valve trim was improperly heat-treated to a strong, brittle condition inappropriate for application in a valve. The aging treatment resulted in a material for which the acceptable room temperature Charpy V-Notch impact strength is as low as 8 J (5 ft-lb).
2. The probable aging condition of H925 was not sufficient to relieve residual stresses that may have resulted from fabrication. An aging treatment at 538°C (1000°F) or over is generally recommended^{6,8} to reduce residual stresses by more than 50%. In areas where stress corrosion cracking may be a problem, the recommended aging temperature is 593°C (1100°F).
3. Residual stresses concentrated at an outside corner provided the probable site for crack initiation. Since the metal was restrained from plastic deformation, when the effective stress exceeded the yield stress, brittle cleavage ensued by the propagation of one crack through the trim, providing stress relief and curtailing growth of the other two cracks.
4. Although 17-4 PH is resistant to stress corrosion cracking, the relatively low-temperature aging may have caused sensitization. The environment of sour gas and recycle water combined with the residual stresses from fabrication to produce stress corrosion cracking, probably accelerated by hydrogen. The local stress and the corroding species cannot be positively identified.

RECOMMENDATIONS

In spite of this failure, the reliability of this valve has shown that with the proper aging condition, 17-4 PH may be an acceptable material for this application. This valve was part of an obsolete group, which had been improperly heat-treated.⁹ In general, these recommendations are offered to prevent future failures of this type:

1. Hardness readings, which should range from 31 to 38 Rockwell C, should be taken before subsequent valves are put in service to assure proper aging at a temperature high enough to relieve residual stresses and increase the ductility and impact strength to acceptable levels.
2. Because of the type of environment and known susceptibility of 17-4 PH stainless steel to stress corrosion cracking in this environment, the valve should be inspected regularly during shutdowns as well as during routine maintenance intervals.

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⁸H. W. Garvin, "Stress Relieving 17-4 PH Stainless Steel by Overaging," pp. 177-79 in *Source Book on Stainless Steels*, American Society for Metals, Metals Park, Ohio, 1976.

⁹D. Canfield, Ft. Lewis SRC Pilot Plant, private communication, July 1978.

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