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### **Engineering Failure Analysis**

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### Failure analysis of cold worked AISI 301 SS diaphragm of gas pump

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| ARTICLE INFO  | A B S T R A C T  |
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| Keywords:<br>Diaphragm<br>Hydrogen pump<br>AISI 301SS<br>Cold work<br>Pit and SCC | Failure analysis was carried out on the ruptured samples of a diaphragm which separate oil and gas in the hydrogen pump. Ferritescope measurement on the fractured diaphragm samples revealed uniform distribution of ferromagnetic phase and it was confirmed as ferritic phase (martensite) by XRD. Substantial ductility in the failed samples was confirmed under quasistatic and dynamic conditions. Pitting and grain boundary attacks were confirmed through microscopic observation. Presence of chloride and alkali metals in the pits, whereas silicon preferentially at the prior austenite grain boundaries were confirmed through Energy Dispersive Spectroscopy (EDS) analysis. Presence of chlorine peak in EDS suggests that chloride ions would have rupture the passive films leading to pit formation. Increasing chloride ion concentration accelerates growth of pits. Growth of pit causes dynamic plastic strain which initiates stress corrosion cracking leading to rupture of the diaphragm. |

#### 1. Introduction

High yield strength and resistance to failure under cyclic stress are primary requirements for a diaphragm material. AISI 301 stainless steel meets these requirements in the cold worked condition [1-3]. Due to TRIP effect, austenite to martensitic phase transformation occurs in this steel as austenite is highly metastable [1-10]. Austenitic stainless steels are known to be susceptible to stress corrosion cracking (SCC) in chloride environment. Takizawa [10] and Chiang et al. [11] have reported increased resistance to SCC up to 30% cold work and decreased resistance to SCC beyond 30% cold work [12-15].

In hydrogen process industry hydrogen is flown through a pump that operates at ambient temperature. The diaphragm in this pump essentially separates oil and gas sides. In the diaphragm, the piston movement on the oil side translates to compression action on the hydrogen side. The pump assembly consists of three diaphragms placed in contact with each other with all the three moving in unison, during operation. The oil pressure is maintained ~5 bar during operation and the pressure difference between the oil side and the gas side is maintained at 1 bar. The diameter and thickness of each diaphragm are 760 mm and 0.5 mm respectively.

Failure of these diaphragms in-service is conventionally determined by monitoring the leakage of process fluid (oil/gas) across the radial slit provided at the central diaphragm location. Pressure sensor are provided to sense these leaks. During the routine check unexpected drop in oil level was observed though the pressure sensors could not sense any appreciable leak. In order to identify the cause of these observations dismantling of pumps was carried out during which it was found that the three diaphragm failed and leaked to a large scale. It was also identified that due to the malfunction of the pressure sensor the leaks could not be detected.

The catastrophic premature failure (at short duration of operation less than 1000 h) and the serious consequences associated with

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**Fig. 1.** Diaphragm from (a) oil side, (b) middle and (c) gas side; Metallographic specimens were extracted from the oil side diaphragm, with a 300 mm long sheet being cut from this diaphragm.

contamination of the gas pipeline with oil warranted a detailed failure analysis of this diaphragm assembly. The systematic studies carried out to identify the reason for failure is detailed in this paper.

#### 2. Examinations and experimental procedure

Photographs of failed diaphragm are shown in Fig. 1a–c. Visual examination of failed diaphragm reveals that diaphragm failed extensively in the oil and gas side as compared to the middle region. From the figures, it is evident that middle diaphragm had one crack which is bounded by pen mark. On the oil side diaphragm, the rupture appeared to have initiated in the circumferential direction (Fig. 1a). The periphery of all the diaphragms was found to be free of cracks.

Fragments of the diaphragms had discontinuous black and brown marks on the surfaces on which grease was applied prior to operation, Fig. 1b. A few secondary cracks were also found close to the fracture locations. The thickness of the fragments was found to be uniform  $\sim$ 0.5 mm, even at locations close to fracture the thickness was found to remain unaltered. Dye Penetrant (DP) inspection carried out on unfragmented portion of the failed diaphragms did not reveal any surface cracks.

#### 3. Results and discussion

#### 3.1. Microstructure

Specimens of dimensions  $10 \times 10 \times 0.5 \text{ mm}^3$  were EDM wire cut from the failed diaphragms and examined under scanning electron microscope (SEM) in different conditions, i.e. as-received, polished, and polished and etched conditions. Etching was carried out electrolytically with 10% oxalic acid. Fig. 2a–d are SE images of the surfaces of the fragments after cleaning with acetone. Fig. 2a–c show the extensive pitting in regions with brown/black patches at different locations. The bright regions seen at the bottom of the deeper pits appear to be deposits or clean surfaces. Fig. 2b and c show multiple cracks propagating from some of the deeper pits. Deep pits, though less numerous, were also seen in regions that were not discoloured. Fig. 2d shows a typical photomicrograph of such a region. Grain boundary attack is also evident in the SE image, marked with square box.

SE images and energy dispersive spectra (EDS) obtained from the prior austenite grain boundary and grain interiors are shown in

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