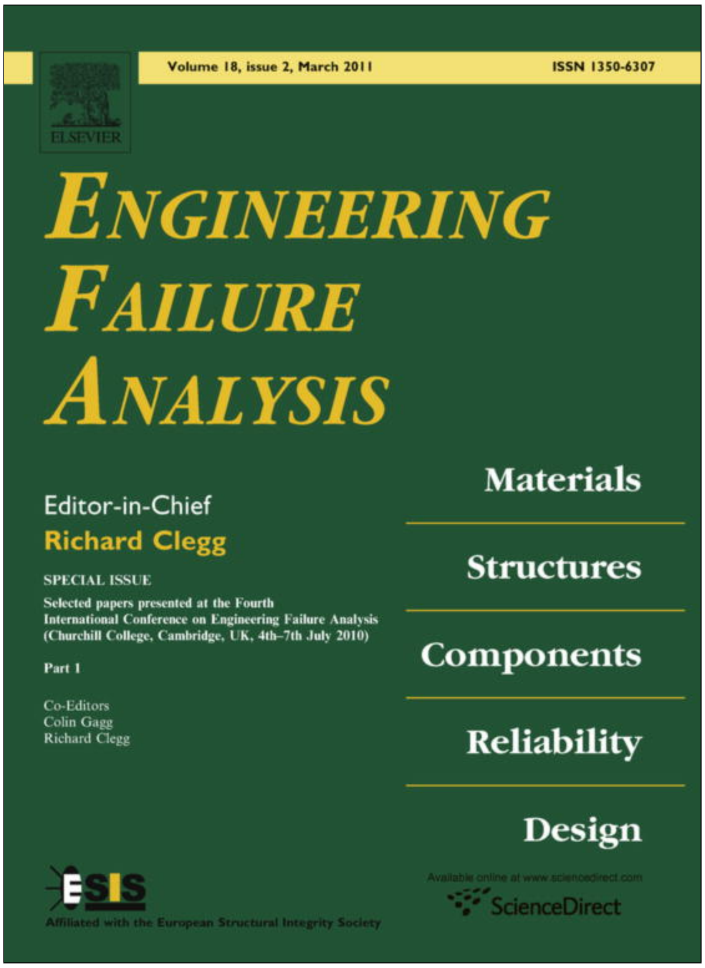
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# Two competing crack growth mechanisms in very high pressures tubes

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ABSTRACT

This work addresses the influence of autofrettage in the occurrence of repeated leaks of thick wall straight and curved 4333M4 steel tubes from a high pressure petrochemical reactor, comprised by straight and curved tubes subjected to autofrettage. Recent leaks in straight tubes are due to fatigue transgranular growth of cracks initiating from machining groves in the inner surface, driven by pressure cycles during many years of normal operation. Experimental measurement of residual stresses verified that high temperature service relieved most of the compressive residual stresses imposed by the autofrettage. Previous failures in curved tubes were found to be due to metallurgical susceptibility to intergranular cracking, related to a phenomenon similar to Temper Embrittlement. Repeating autofrettage to mitigate fatigue failures would reproduce the deformation mechanisms that favored intergranular cracking of the tubes. Recently developed life extension strategies include early detection of leaks by acoustic emission and an improved manufacturing method of replacement bends.



## 1. Introduction

This study was carried out after two fatigue failures of thick wall straight steel tubes, used in a jacket type high pressure polymerization reactor. It is composed by individual tubes, bent to a U-bends. The reactor has several curved steel tubes, of diameters 113 mm (outer diameter – OD) and 51 mm (inner diameter – ID). Along the whole length of the reactor, this inner tube transporting polymer is surrounded by an outer tube, between these, refrigerating treated water circulates. The diameter of each curve is around 2.4 m; the length of the tract up to the thread is of 4.2 m. Within the tubes ethylene circulates in a polymerization stage. These fatigue failures follow other failures in the same reactor, but of an intergranular type, concentrated in the curved tracts, between 2002 and 2005 [1]. These failures involve unexpected plant stops, production losses and maintenance costs. For about 20 years the reactor worked to a pressure of around 250 MPa, with approximately 45 stop and run cycles per year that is to say about 900 pressure cycles. Process valves produce low frequency fluid pulsations (1/25 Hz) of large amplitude (approx. 50 MPa). The reactor also suffers high frequency (20 Hz) and low amplitude pulsations due to the operation of a six cylinder compressor that rotates at 200 rpm. Tubés temperature is about 300 °C.

The chemical, mechanical and dimensional specifications correspond to Timken 4333M4 tubes, see Tables 1 and 2 [2]. Elongation is in one case below tolerance. Hardness values in all failed curves compared well with specifications for Nominal fittings AISI 4340 [3]. This steel has high strength, due to high contents of C, Mn, Cr and Mo, and good ductility, due to a 2% Ni.

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**Table 1**
Chemical composition of tubes Timken in polymer reactor.

| Material | C | Mn | P | Si | Cr | Ni | Mo |
|---|---|---|---|---|---|---|---|
| Fatigued tube 1 | 0.33 | 0.76 | 0.01 | 0.26 | 0.69 | 1.81 | 0.43 |
| Fatigued tube 2 | 0.38 | 0.98 | 0.012 | 0.21 | 0.79 | 2.15 | 0.74 |
| Old curve 3 | 0.31 | 0.87 | 0.012 | 0.34 | 0.65 | 1.82 | 0.43 |
| New tube 4 | 0.34 | 0.86 | 0.012 | 0.25 | 0.69 | 1.85 | 0.49 |
| Curve 5 | 0.35 | 1.00 | | | 0.70 | 2.0 | 0.50 |
| Specification | 0.30–0.38 | 0.7–1.0 | 0.015 max | 0.20–0.35 | 0.7–0.9 | 1.65–2.0 | 0.35–0.45 |

**Table 2**
Mechanical properties of tubes Timken in polymer reactor.

| Tube | Yield strength (MPa) | UTS (MPa) | Elongation (%) | Area red (%) |
|---|---|---|---|---|
| Curve 5 | 1095 | 1188 | 10.5 | 57 |
| Specification | 980 min | 1120 min | 14 min | 45 min |

These tubes are manufactured by electric arc, with degassing and deoxidation processes, then they are bent and subjected to quench and tempering heat treatments.

Manufacturer data indicates that after the curving process the tubes are heat treated at 490 °C. The curved tubes are afterwards internally pressurized in order to assure plastic deformation of the material near the ID. This creates a circumferential residual stress field that is compressive near the ID and tensile near the OD. This is called autofrettage. The autofrettage pressure is fine tuned according to the actual yield strength of each batch of tubes (for these tubes, the pressure is around 724 MPa).

## 2. Fractographic analyses and assessment of residual stress

Thickness and outer diameter are within tolerance. It is observed that due to the rolling process from the outside, thickness variations are translated into variations of the inner diameter, the outer diameter being within very good dimensional tolerances. The manufacturing process did not produce dimensional variations above specified tolerances. As already mentioned, both tubes failed due to the propagation of longitudinal cracks initiated at the inner surface of the straight section of the tubes. No secondary cracks were detected. Fig. 1 shows a sample taken from the first fatigue crack; note the crack path along the inner surface of the thick walled tube. Fig. 2 shows the surface of both identical fatigue cracks, after cryogenic

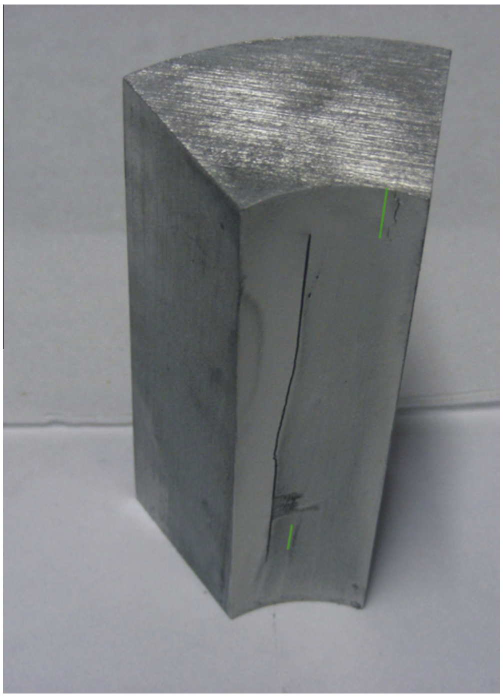

**Fig. 1.** Sample taken from first fatigue crack, crack path along inner surface of thick walled tube.

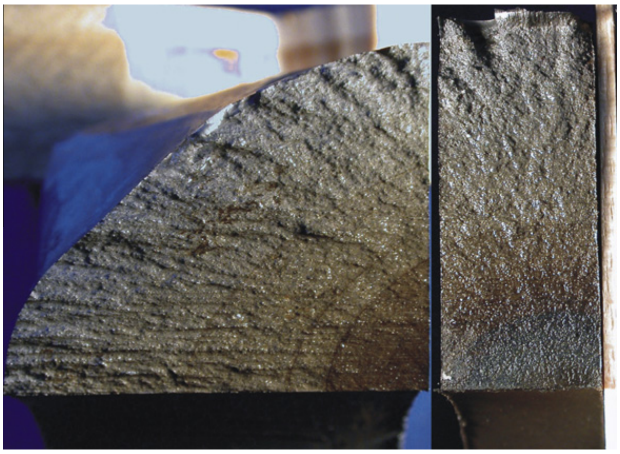

**Fig. 2.** Surface of first fatigue crack, after cryogenic opening and cleaning, note initiation from inner surface (bottom of photo).

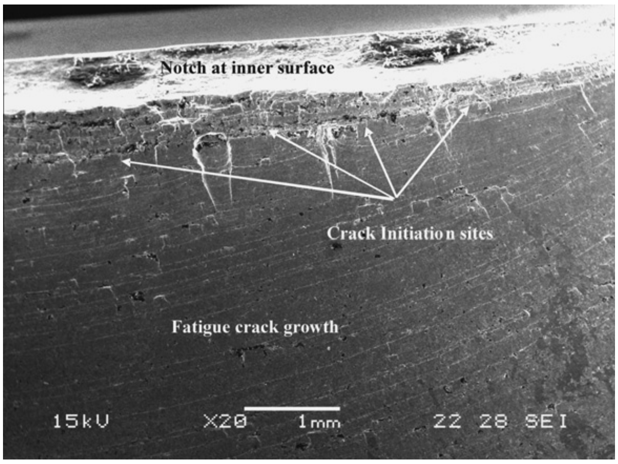


**Fig. 3.** Crack growth from preexisting defect (20×).

opening and cleaning. Note initiation from the inner surface and beach marks, clearly denoting successive crack fronts during in-service propagation.

Fig. 3 (20×) shows a scanning electron microscope (SEM) fractography. The crack shows typical propagation from multiple initiation sites, at the bottom of a notch at the inner surface of the pipe wall. Most relevant is the distance of the multiple initiation sites to the bottom of the notch, there is a 0.3 mm deep band from which the fatigue cracks start. Fig. 4 shows the shape of typical preexisting notches at the inner surface of tube (150×, 2000×). These machining notches are normal for this type of component. The micrographs show the acicular characteristic of a tempered martensitic microstructure. Note that not only the preexisting notches at the inner surface create stress raisers, some inclusions at the bottom of these notches would become very effective fatigue initiation sites.

One possible cause for the observed failures is that long service times at high service temperatures would severely reduce the compressive circumferential residual stresses introduced during manufacturing with the autofrettage process. Residual stress analyses at bends were discussed in a previous work by the authors [4]. Fig. 5 shows the measured circumferential residual stresses along the pipe thickness. Maximum circumferential residual stresses near the inner pipe surface are up to 400 MPa in compression, in the plane of the curve. Tensile maximum is only 70 MPa. But residual stress measured at a 90° position along the pipe perimeter is much lower. In any case, this experimental evidence is not conclusive as whether or not the residual stress field has been destroyed by service temperature.

## 3. Previous assessment of intergranular failures

Failures at bends are characterized by intergranular and branched cracks propagation. Before 2004, cracks initiated at the outer surface (water side). Fig. 6 shows a typical H-shaped branched crack, as seen when testing with black on white magnetic particles the outer surface of the thick walled tube. Subsequent ultrasonic mapping allowed to recognize typical crack branching and propagation in the pipe wall, with many smaller secondary cracks. Stresses due to internal pressure, pipe sup-

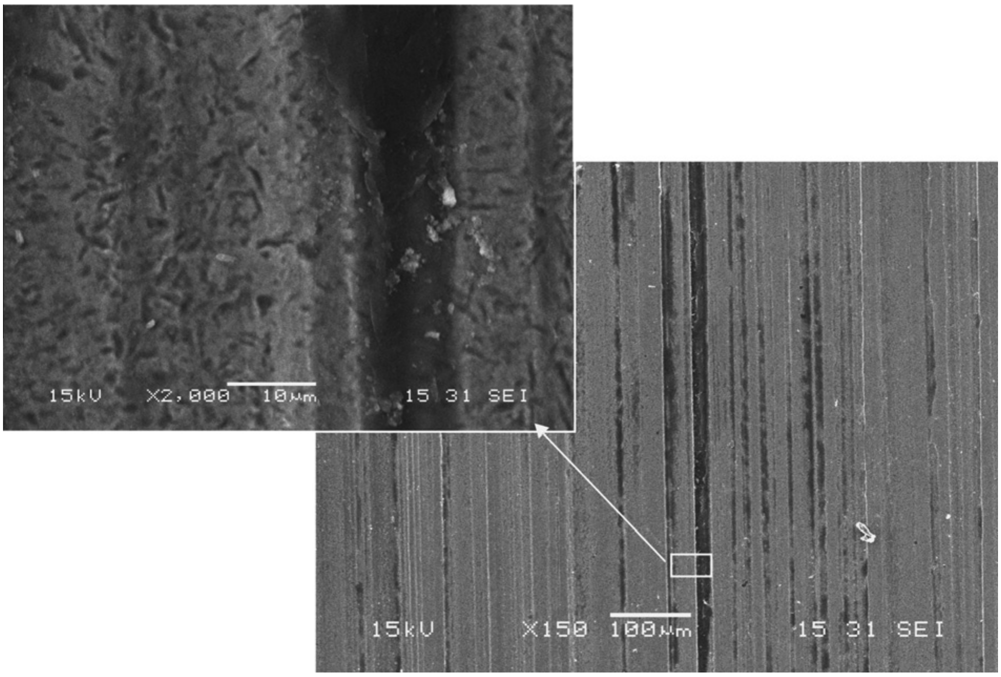


**Fig. 4.** Preexisting notches at inner surface of tube (150×, 2000×).

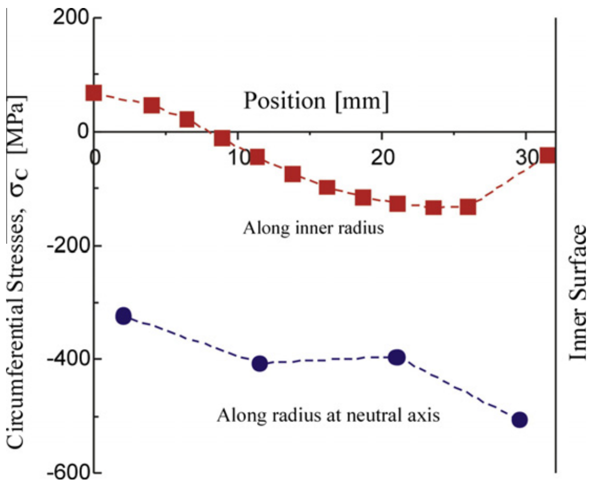


**Fig. 5.** Circumferential residual stresses.

port conditions and in-service expansions were modeled, and found to be high enough to justify the required crack driving forces for Stress Corrosion Cracking. Alkaline agent, forming Na(OH), was eventually replaced by morpholine. However, subsequent failures with similar growth characteristics initiated in the inner surface and in the mid-thickness of the tube. These proved that previous assumptions did not fully explain the failure mechanism. Examples of Fig. 7a and b correspond to (a) crack in curve 3 old (2004), initiated at the inner surface; and (b) curve 3 new (2004), initiated at mid-thickness and failed after less than a month in a replacement tube. These last failures prompted a study to determine the weaknesses of the material that eventually favored the occurrence of such failures, in order to define and implement effective mitigation measures. These studies are presented in a previous article by the authors [5]. Transgranular Tempered Martensite Embrittlement (TME) was detected at test temperatures close to the ductile–brittle transition temperature. The phenomenon was related to the formation of coarse carbides at the martensite lath boundaries [6], nevertheless some authors do not agree with this theory and attribute transgranular TME to the decomposition of retained austenite instead of the carbide coarsening [7]. The intergranular type of TME has been observed generally at test temperatures below the transition temperature and was associated with the combined action of coarse carbides and impurities at the prior austenite grain boundaries. If the grain boundaries act as the slip barriers, the blocked slip bands can induce the crack nucleation at the grain boundaries [8].

The possible incidence of embrittlement effects was investigated by means of Charpy impact tests at room temperature on ex-service samples. Freshly broken fracture surfaces were then inspected in the SEM. Fig. 8 shows a typical fracture sur-

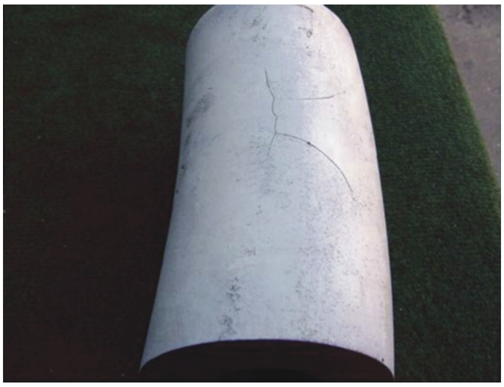

**Fig. 6.** Typical H-shaped, branched crack, as seen in the outer surface of the thick walled tube.

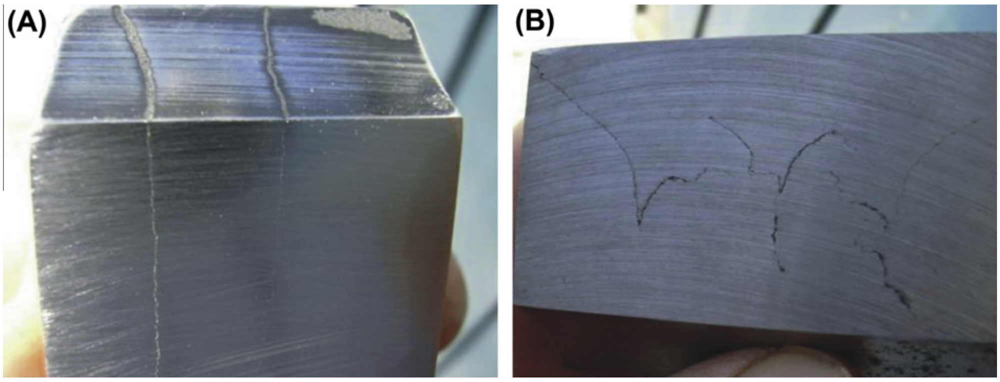


**Fig. 7.** Cracks corresponding to different failures, initiated: (a) at the inner surface and (b) at mid-thickness.

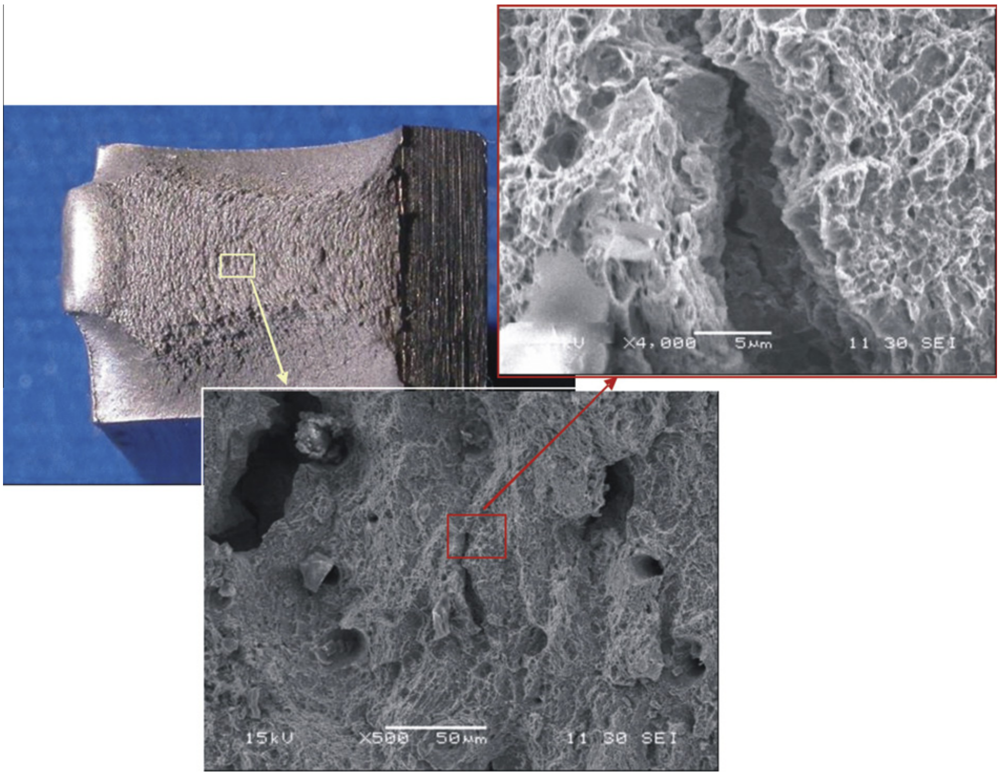


**Fig. 8.** Typical fracture surface of a specimen from a cracked curve (250×, 2000×).

**Table 3**
Room temperature Charpy impact test results.

| Specimen | Position | Subsize 7 × 10 (J) | Std 10 × 10 (J) |
|---|---|---|---|
| Uncracked tube | Straight | 55 | 78 |
| | Curve | 57 | 81 |
| Curve 5 | Near crack | 30 | 42 |
| New 3 | Near crack | 52 | 74 |

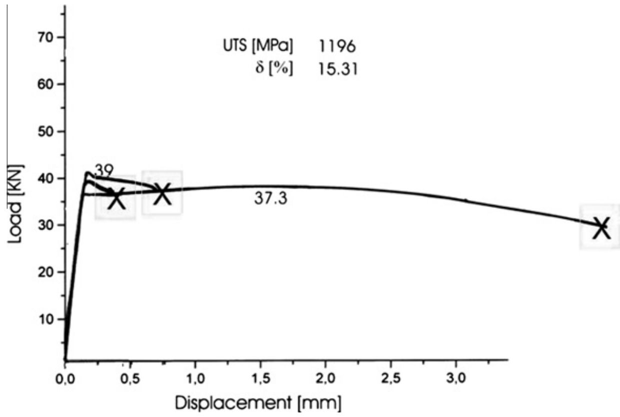


**Fig. 9.** Tensile test curves of the material in its original state, and with thermal–mechanical embrittlement.

face of a specimen from a cracked curve. All specimens show well developed shear lips close to three free surfaces, which justify high values of impact energy, see Table 3, and which are apparently unaffected by any embrittlement. In the 30% inner area, however, the fracture propagated in a wavy pattern, that reveals a specific crack path related to the weakest microstructural features. The upper inlet in Fig. 8 (4000×) shows a micro void ductile fracture mechanism, within many secondary cracks produced by the impact.

The influence that previous plastic deformation exercises upon the toughness of the tempered martensite was evaluated by subjecting the martensitic structure of the tubes to plastic deformation and temperatures similar to those found in normal service, and evaluating their influence on mechanical properties. Tensile specimens were deformed 3% and 5%, then subjected to temperatures of 300, 325, 350 and 375 °C during 24 h, and finally broken. Tensile tests with notched specimens were also carried out. Fig. 9 shows load–deformation curves for the material in its original state (specimen #1). The material shows low strain hardening, what is translated in an ultimate tensile stress (UTS) very close to the yielding. The specimens deformed 3% and 5% present larger yield stress, as expected, but also showed some unexpected behaviour.

For all maintenance temperatures, when reaching the yielding load the specimens destabilized, that is to say, yielding coincides with the peak load and in that point necking takes place. The decrease of load evidences that the plastic deformation of the specimens is minimum, and all the deformation is due to necking. This behaviour would be critical if for some reason the material is imposed to a restriction of plastic flow, as happens in the middle area of the cross section. When imposing a restriction to necking the specimens break practically without plastic deformation (0.8%). It is possible to conclude that the evidenced phenomenon of embrittlement is due to a combination of both parameters: plastic deformation and maintenance to temperature.

A fractographic characterization was carried out after testing, in samples from original and embrittled materials. Fracture surfaces in original material show a rosette fracture type, typical in steels subjected to tension. The fibrous central area is where the fracture originates; it has an appearance of fibres at random with micro voids in the whole surface. The fracture surfaces of the embrittled samples do not present marked differences related with the amount of deformation or the maintenance temperature. In all cases the fracture continues being of the rosette type. These deformed samples have a radial area with a great quantity of longitudinal cracks that define a more irregular fracture path, with more crests and valleys. Fracture surfaces change in the case of the notched tensile specimens. Fig. 10 shows the fracture surface of specimen #9, with a previous 5% plastic deformation and a temperature of 325 °C during 24 h. Circumferential waves are observed in the propagation direction, inside the intergranular cracking, microvoids have been observed.

## 4. Discussion of results

Depth of machining marks, around 10 μm, was not enough by themselves to initiate the cracks. Machining grooves and nonmetallic inclusions were combined to form longitudinal defects up to 0.3 mm defects in the ID, from where fatigue cracks initiated. In both cracks investigated, a zone immediately underneath the groove is distinguished by fatigue propagation of

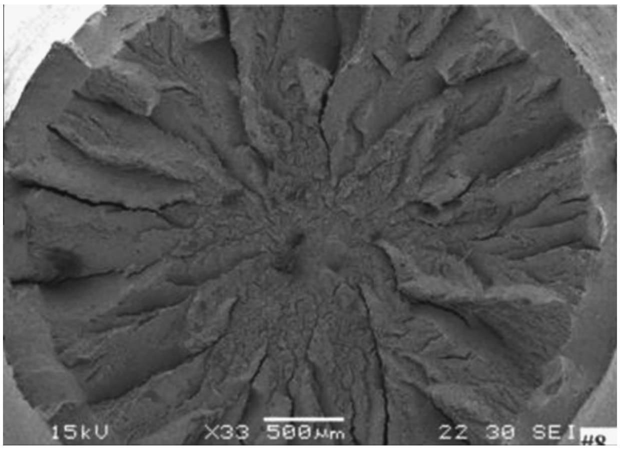


**Fig. 10.** Typical rosette type fracture surfaces in notched embrittled tensile specimens.

the crack towards the outside of the tube, see Figs. 1 and 2. This zone is a manufacture defect of the material, probably a lamination defect. River patterns indicated by arrows in Fig. 3 reflect changes of initiation planes from this preexisting defect.

The material presents high density of inhomogeneities that create susceptible zones for the nucleation and propagation of cracks. The mechanism of propagation by fatigue rarely leaves in martensitic steel the indicative microscopic striations during crack advance, but clear beach marks show the evolution of the crack, see Figs. 2 and 3. Fracture surfaces present squashed regions, Fig. 3, that allow considering that the cyclic component of stresses presents a compressive part.

SEM inspection allowed counting beach marks following three radial paths on the crack surfaces of each fracture. They average 84 beach marks, which were associated to the start–stop cycles of the reactor. Operation pressure cycles are shown in Table 4. There are approximately 1185 stop start cycles, programmed and not programmed. On this basis, it was possible to go backwards and evaluate the most probable date for crack initiation; this was 2 years before the leak was detected.

The fatigue damage is due to the variations of polymer pressure inside the tubes. Cyclic design stresses are below fatigue threshold. As only the positive parts of stress cycles contribute to fatigue growth [9], crack initiation could be related to a reduction in compressive circumferential stress at the ID, intentionally created by the manufacturing autofrettage process. Neither the fractographic evidence nor the residual stress measurements are conclusive in confirming this condition. The presence of initial defects justifies the failures, probably activated by an event of over pressure. Therefore it is believed that the reactor is not yet near the end of its service life.

These results helped define the strategies for life extension and reduction of operation costs. The main cost of each failure has been up to now related to the time required to detect where the leak is. Therefore, an early detection system has been tested using the acoustic emission technique. Probes in the outer walls of the jacket tubes hare being placed, which will allow detecting any leak and define its position.

It is now clear that straight and curved tubes have different failure modes and therefore should be replaced with different criteria. Intergranular degradation of the curved tubes has been shown to be due to the plastic deformations during curving. Work with the manufacturer concluded that local deformations could be reduced by improving the curving process. New curves will be provided with a better controlled mechanical bending, that will lead to more uniform and controlled amount of plastic strain in the complete length of each curve. Failed straight pipes will be replaced with autofretted pipes, and straight tubes still in use may in turn be recovered with a repeated autofrettage.

## 5. Conclusions

This work addresses the influence of autofrettage in the occurrence of repeated leaks of thick wall steel tubes from a petrochemical reactor made of 4333M4 steel, operating at very high pressure (around 250 MPa). Experimental analyses of two recent fatigue failures were presented, which are the result of transgranular growth of cracks initiating from machining groves in the inner surface of the tubes, driven by pressure cycles during many years of normal operation. Experimental

**Table 4**
Assessment of load cycles suffered by reactor tubes.

| Cyclic loads | Pressure (Mpa) | Frequency | | Fatigue life | |
|---|---|---|---|---|---|
| | | Hz | Cycles per year | Years | Cycles |
| Stop–start | 230 | – | 45 | 26 | 1170 |
| Let down bump valve | 50 | 0.04 | 1,261,440 | 26 | 32,797,440 |
| Compressor | No data | 20 | 630 million | 26 | 1.64*E*+10 |

measurement of residual stresses helped verify that high temperature service relieved most of the compressive residual stresses imposed by the autofrettage.

When addressing the convenience of repeating autofrettage as a means to avoid fatigue crack initiation, careful attention had to be given to previous leaks occurred in this reactor. Failures initiated in the outer surface due to metallurgical susceptibility to intergranular cracking were characterized by strongly branched, mostly circumferential multiple intergranular cracks. In a previous work by the authors, susceptibility to intergranular cracking was experimentally assessed by recreating conditions of embrittlement by thermal treatments and tensile testing. Under the conditions of previous plastic deformation due to bending and autofrettage it was possible to recreate intergranular embrittlement at service temperatures, a phenomenon similar to Temper Embrittlement.

The forming process created localized yielding and large longitudinal residual stresses. Recovery measures, mostly relying on thermal treatments, would possibly mitigate failures due to intergranular damage. But repeating autofrettage to mitigate fatigue failures would reproduce the deformation mechanisms that favored intergranular cracking of these tubes. Straight and curved tubes have different failure modes and therefore should be replaced with different criteria. Local deformations in curves could be reduced by improving the curving process. New curves will be provided with a better controlled mechanical bending, that will lead to more uniform and controlled amount of plastic strain in the complete length of each curve. Failed straight pipes will be replaced with autofretted pipes, and straight tubes still in use may in turn be recovered with a repeated autofrettage. In order to reduce the costs of future leaks, early detection of leaks by acoustic emission is being implemented with probes located in the outer surfaces of the jacket tubes.

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