In situ metallography as non-destructive test to analyze the microstructural damage in the petrochemical industry

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Abstract
In situ metallography is required when the metallurgical damage must be evaluated on industrial equipment. The metallurgical damage to be evaluated in metallic equipment could be classified as: creep, graphitization, precipitation of sigma phase, grain growth, etc. There is also the need of in situ metallography when repairs of equipment are been carrying out in order to make sure that weldments of stainless steel are free of precipitation of second phases or, if it is required the inspection of the microstructure, to meet the microstructural requirements of the standard. This paper shows the results of field work using in situ metallography and evidence of the reliability of the results for representing the bulk microstructure of the metallic component.

Keywords: In situ metallography, microstructure, damage mechanism, tubes, steel

1. Background

Petrochemical plants in Mexico have been operating for more than 30 years and there is a need to monitor the metallurgical state of the industrial equipment, especially in equipment that works at high temperature or that has been exposed to fire [1]. The need of checking the microstructural state of the industrial materials arises from the fact that microstructure strongly influences the mechanical and corrosion properties of materials [2,3].
The traditional metallographic preparation, where the specimen must be cut, mounted, grinded, polished and etched, has the disadvantage that the metallic component has to be destroyed [3], but in petrochemical plants (and in other industries also) there is a necessity to monitor the metallurgical state of the microstructure without destroying the components of the equipment, therefore in situ metallography is a suitable technique for that purpose [3-5].

2. Objective

The main objective of this paper is to show evidence of the reliability of the field results using in situ metallography for representing the bulk microstructure of the metallic component and to show results of field work in order to illustrate the importance of this technique for the petrochemical industry.

3. Experimental methodology

In this paper it is described the application of non-destructive metallography in petrochemical equipment to evaluate qualitatively one of the aforementioned damage mechanisms and in order to be sure that in situ metallography is representative of the bulk

microstructure. A creep failed tube was cut in the laboratory and then a non-destructive metallography was carried out in two zones, one damaged by creep and another away from the damage. The failed tube is shown in Figure 1. As it can be seen in the figure, the tube shows bulging due to overheating damage; two samples were cut, mounted and prepared for traditional metallographic analysis on the middle plane (in the center of the material) and observed using a NIKON EPIPHOT 200 optical microscope [3]. Sample denominated as Zone 1 was taken in the bulging zone near the failure and the Zone A, which was prepared for in situ metallography. At the other hand, a sample denominated as Zone 2 was taken away the bulging, where no damage could be seen. The sample denominated as Zone B was prepared for in situ metallography in the location shown in Figure 1. The in situ metallography microstructures were observed using a portable Struers WF 10X DIN/18 optical microscope.

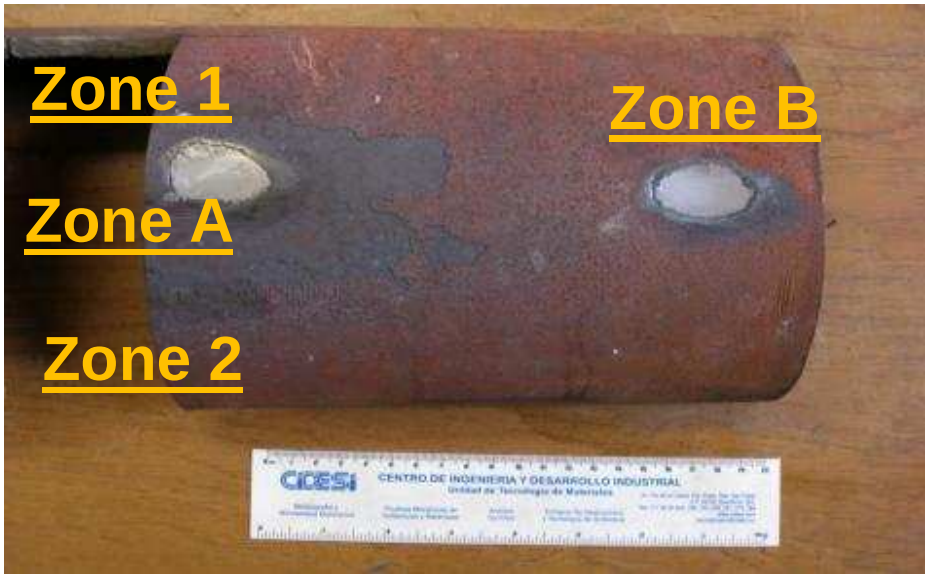

Figure 1. The image shows the failed tube, where the samples and the in situ metallographies were taken. It can be seen the location of Zone A, Zone B, Zone 1, and Zone 2.

All microstructures were revealed using Vilella's reactive. The samples prepared for traditional metallographic analysis were observed at 500X (Zones 1 and 2), but in situ metallography microstructures (Zones A and B) were observed at 400X. The 500X magnification for traditional samples was chosen because it was the nearest to the magnification of the CIDESI's portable microscope (400X). Details of the sample preparation in the laboratory and the sample preparation for in situ metallography can be found elsewhere [3-5].

4. Results and Discussion

The microstructures observed using laboratory microscope are shown in Figures 2 (Zone 1) and 3 (Zone 2). The microstructures observed using in situ metallography technique are shown in Figures 4 (Zone A) and 5 (Zone B).

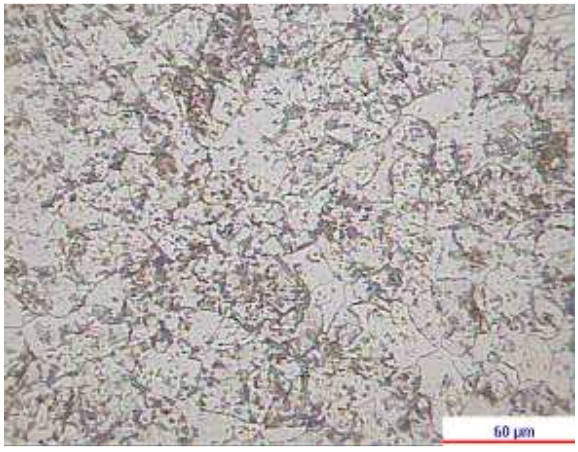

Figure 2. The micrograph shows at 500X the microstructure of the damaged tube in Zone 1.

Figure 3. The micrograph shows at 500X the microstructure of the damaged tube in Zone 2.

Figure 2 shows the typical microstructure of an overheated steel tube, which consisted of elongated pearlite and carbides precipitated on the grain boundary (this microstructure helps to understand the cause of the failure of this tube, nevertheless, it is not the aim of this paper to discuss the failure due to overheating). Here must be mentioned that the dark regions of the microstructure are not pearlite regions; one must keep in mind that the microstructure was taken very near the failure due to overheating and this tube suffered thinning, therefore the middle plane is near the external surface where a layer of fire residuals were deposited; normally this deposits layer react with the steel of the tube leading to such appearance.

Figure 3 shows the microstructure away from the bulging of the tube. This microstructure shows evidence of no strong carbide precipitation in the boundary between phases. In this case the pearlite cannot be distinguished because the sample was etched using Vilellas etching during the same time as in the microstructure of Zone 1. This is due to the fact that longer etching times in Zone 1 led to overetching and no microstructure was revealed. Besides, it is important to mention that in case of microstructure of Figure 3 the boundary showed on the micrograph was delimitated by the etching and not by carbide precipitation

as it is the case in Figure 2. The important fact of this microstructure is that the phases are not elongated.

Figure 4. The micrograph shows at 400X the microstructure observed using in situ metallography in zone 1.

Figure 5. The micrograph shows at 400X the microstructure observed using in situ metallography in zone 2.

Figure 4 shows the in situ metallography microstructure of Zone 1, which corresponds to the laboratory microstructure of the Zone A. Evidence of carbide precipitation in the grain boundary can be seen, which delimitated very clear the grains, and no evidence of pearlite bands. In the micrograph it can be seen that the grains are elongated as in Figure 2.

Figure 5 shows the in situ metallography microstructure of Zone 2, which corresponds to the laboratory microstructure of Zone B. It can be seen no evidence of carbide precipitation because no limited phases are delimited, therefore no elongated phases appear.

The comparisons between Figure 2 and Figure 4 and between Figure 3 and Figure 5 indicate similar features in the microstructures. This fact leads to state that results of in situ metallography are comparable qualitatively with results using traditional metallography using conventional laboratory techniques, therefore, results using in situ metallography are qualitatively representative of the microstructure.

One of the advantages of in situ metallography is that there is no need to destroy the metallic component in order to get metallurgical information, therefore the application of this technique can be considered as nondestructive [6,7]. Besides, to evaluate creep damage in petrochemical plants in situ metallography has been applied for reveling the kind of material of an equipment. Figure 6 shows personal of CIDESI carrying out in situ metallography on an equipment, where no data about the type of gray iron was available. Figure 7 shows the resulting microstructure.

Figure 6. Here it is shown the in situ metallography on a plant equipment.

Figure 7. The micrograph shows a gray iron with E type graphite.

In Figure 8 boiler‘s tubes are shown. The boiler was out of operation for maintenance and in situ metallography. Figure 9 shows the result of the in situ metallography. The microstructure consisted of ferrite and pearlite; there was evidence of degradation of the microstructure.

Figure 8. Tubes of a boiler during maintenance.

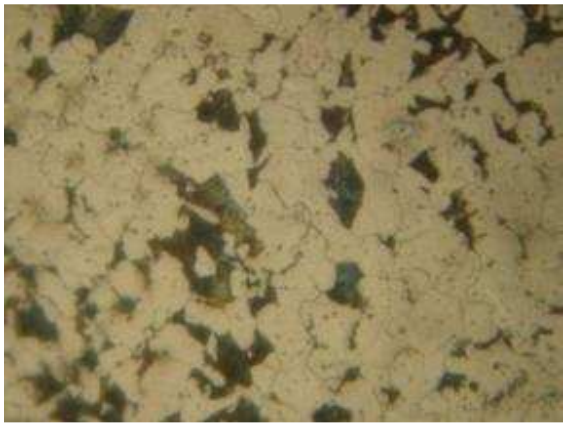

Figure 9. Microstructure of the Boiler's tubes.

These results are examples of the normal practice of maintenance in boilers tube. This practice is carried out for periodical monitoring of the metallurgical degradation of the microstructure in the tube [7]. The aim of this practice is to ensure the mechanical properties of the tubes to withstand the internal pressure for preventing failure [7]. Nevertheless, it must be mentioned that located overheating of the tubes accelerates the damage of the microstructure leading to failure and stops for repairing of the equipment.

Another example of the application of this technique is the following: an equipment in a petrochemical plant was heated during a long period of time in one side (no more information about this case was available). In order to evaluate the metallurgical damage in situ metallography was carried out.

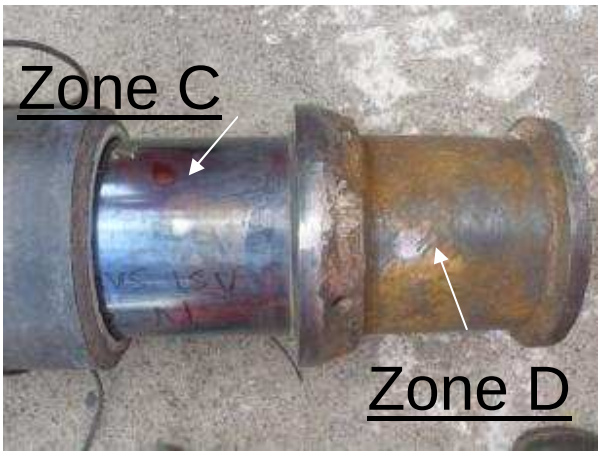

Figure 10. The image shows the zones (Zones C and D) where the in situ metallography was carried out in the equipment.

In this case, it can be seen that grains in Zone D are bigger than grains in Zone C, indicated grain growth in Zone D, where heating had taken place. The grain growth was found in a qualitative way by comparing the microstructure at the same magnifications. However, no evidence of sigma phase precipitation was found. One example of a damaged microstructure by sigma phase precipitation is shown in Figure 13. The equipment shown in Figure 13 failed suddenly after 15 years of service and the operation had to be stopped for repairing. This stop might have been avoided if in situ metallography had been carried out during maintenance.

Figure 13. The image shows a damaged equipment.

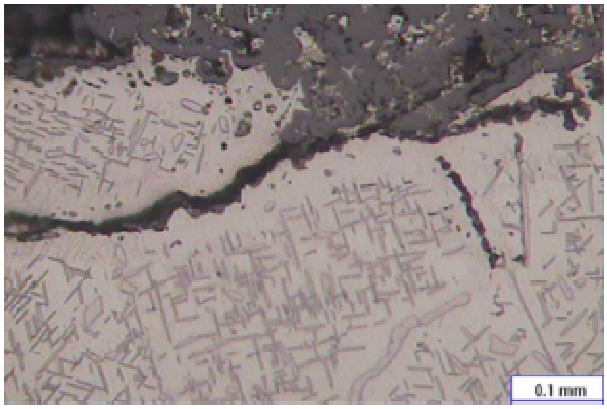

Figure 14. The image shows the microstructure of the equipment shown in Figure 13. It can be seen sigma phase precipitation.

A valve's cover had failed in a sudden way. As first approximation in situ metallography was carried out in order to find if the material of the valve's cover was correct.

Figure 13. The image shows the failed valve's cover.

Figure 14. The micrograph shows the microstructure of the failed component, which consisted of gray iron.

Using in situ metallography the microstructure of the gray iron was revealed and it consisted of graphite flakes and a matrix of pearlite, but also was found evidence of a bright phase. Using other experimental techniques it was discovered that this bright phase consisted of steadite (Fe_3P).

5. Conclusions

In order to find out the reliability in a qualitative way, a comparison between traditional metallographic preparation and in situ metallography was carried out in a by overheating failed tube. The results of the comparison of the microstructures showed quantitatively the same characteristics of the microstructure.

Because the petrochemical equipment is mainly manufactured of carbon steel or austenitic stainless steel, in situ metallography is applied in order to check damage mechanism in ferrous alloys such as: graphitization, degradation of pearlite, creep, decarburization, grain coarsening (grain growth), intergranular corrosion, carbide precipitation, and precipitation of sigma phase in stainless steels. In situ metallography was applied as a non-destructive method to analyze the microstructure of metallic equipment in order to assess the metallurgical integrity of equipment in petrochemical industry and some examples are shown in this paper.

In situ metallography can be applied as a first approximation for a failed component, as Figure 14 showed, but this technique is not enough to find failure's cause out, but thoroughly failure analysis must be carried out.

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