Failure Analysis of Jet Turbine Engine High Pressure Nozzle Material Made of Cobalt Based Super Alloy

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Abstract: In this study, we carried out the failure analysis based on metallographic research of a cobalt superalloy material which had been used in jet turbine engine. The failure analysis was made by chemical composition analysis, hardness test and microstructural researches after metallographic sample preparation on the material which had operated about 30000 hours and damaged. Macro cracks which can be seen by visual inspection have been detected on the cobalt based superalloy nozzle material's some surfaces which had exposed to high temperature and high pressure fuel-air mixture. After the metallographic sample preparation and optical and electron microscope investigation, it has been determined that the main reason of the failure is coarse and continuous morphology M_7C_3 carbide precipitations on the grain boundaries and as a result of that, stress corrosion cracking has moved through that grain boundaries which have been embrittled and weakened against high temperature corrosion. Also, after consideration of the chemical composition on the damaged area of material, it has been detected that, owing to the usage of carbon content fuel during operation, carbon diffusion at high temperature into the material had occurred. So, with high carbon content in the material, the type and morphology of the carbides had changed and caused the failure.

Keywords: Failure analysis, jet engine and turbine, nozzle, cobalt base superalloy, carbide transformation, stress corrosion, microstructure, optical microscopy.

1. INTRODUCTION

Nickel and cobalt based superalloys are the materials which have been widely used in both aerospace industry and also gas turbines. Their excellent creep resistance, high temperature corrosion resistance and also having higher strength at elevated temperatures make cobalt based super alloys more profitable in comparison with nickel based superalloys [1]. In this sense, cobalt based superalloys are widely used in some critical parts of jet engines. One of these critical materials is high temperature nozzle. The task of this material which is used as first stage high pressure nozzle in jet engines and power turbines is conveying the hot gas flow that released from the combustion chamber most efficiently and with best angle through the rotating parts of turbine. Therefore, more than mechanical stresses, this nozzle is exposed to thermal loadings and also bending stresses due to the gasses which have high temperature and pressure. In this case, the possible material failure mechanisms which can be occurred during these operation conditions are creep, creep-fatigue, thermal fatigue, oxidation and high temperature corrosion [2].

The most important factor that determines the performance of the nozzles made of cobalt based superalloys is their microstructures. The microstructures of these materials might be changed during the operation because of high temperatures. The phases in the microstructure, morphologies of them and the regional chemical compositions can be changed. Therefore, the failure analysis is carried out in the foreseen lifetime of the material and reparation or renewal decision is made according to the analysis. The basic method for failure analysis of these type of cobalt based superalloys is metallography. The sample prepared by metallographic sample preparation processes is subjected to the optical microscopy and/or electron microscopy investigation. As a result of microscopic investigation acting like a detective searching a crime, according to the failure or crack structure and according to the phase changes in the microstructure of material, the failure mechanism is determined.

The aim of that study is to determine the reason of the failure on the first stage high pressure nozzle material which had operated approximately 30,000 hours while it had been predicted to operate for 70,000 hours. In addition to that, another aim of that study is to determine the correct metallographic sample preparation processes of cobalt based superalloys. By carrying out optical microscopy and electron microscopy investigations, determination of the failure mechanism on the nozzle and according to the results, improving the service life of the material are the main targets of that study.

2. MATERIALS AND METHODS

The material on which failure analysis has been carried out is a cobalt based superalloy which had operated for 30,000 hours inside the General Electric CF6-50 high by-pass turbofan engine as first stage high pressure conveying nozzle and it has been shown on Figure 1.

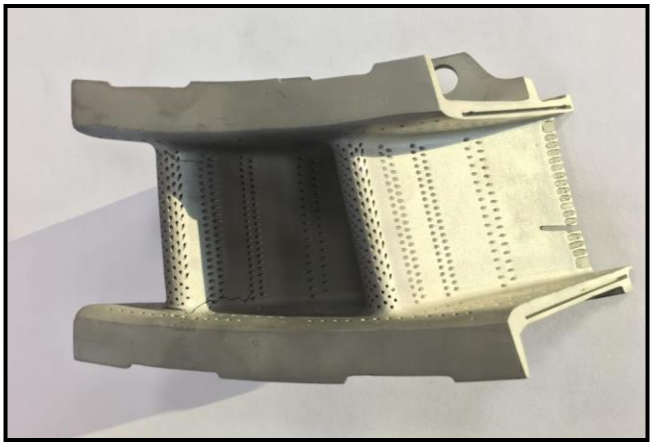

Figure 1. First stage high pressure nozzle

2.1. Sampling

After visual inspection, sampling was carried out from the macro crack zones and also non-damaged parts for comparison. The sampling zones of the sample and the names of zones were shown on Figure 2.

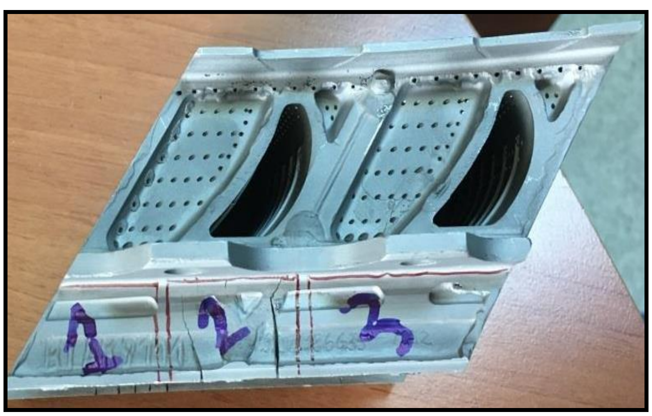

Figure 2. Sampling zones

The cutting process for sampling step of metallography was carried out by using Metacut 251 abrasive cutting machine which has SiC cutter disk and liquid cooling system in Istanbul Gedik University Metallurgical and Materials Engineering Laboratory.

2.2. Hot Molding

Hot molding method was preferred in order to both make cut parts hand-held and inserted into neatness for all next steps which are grinding-polishing, etching and microscopy. Ecopress 50 Hot Molding Machine and bakalite and acrylic powders were used for molding process. The temperature was 190^0C, the pressure was 130 bar and the duration was 3 minutes for sintering and 3 minutes for cooling.

2.3. Grinding – Polishing

After molding, grinding and polishing processes were applied to the samples by Metkon Forcipol 2V machine which is located in Istanbul Gedik University Metallurgical and Materials Engineering Laboratory. SiC grinding disks were used as abrasives during grinding process. The rotational speed was 300 rpm during both grinding and polishing. For polishing, 0.25 µm mono crystalline diamond suspension and diamond based lubricant were used.

2.4. Etching

For microstructural analysis with optical microscope; from optic physics and inorganics chemistry aspects, the requirement is creating metallurgical contrast on the surface to be investigated. In order to actualize it, a chemical called etchant is used.

In order to carry out optical microscopy investigation, etching process was applied to the polished samples with convenient etchants and in optimum etching durations. We applied the both chemical etching and electrolytic etching methods.

For chemical etching, Kallings No:2 etchant was prepared with 5 gr $CuCl_2$, 100 ml hydrochloric acid (HCl) and 100 ml ethyl alcohol (ethanol). For electrolytic etching, a solution which includes 10 ml HCl, 10 gr CrO_3 and 100 ml distilled water was prepared. The sample was attached to the positive pole of a direct current power supply as anode and 304 stainless steel as cathode to the negative pole. Both anode and cathode were submerged into the etchant solution and 6 V voltage has been applied for 10 seconds duration. Immediately after etching, the sample was washed thoroughly with ethyl alcohol and water and dried with warm air flow.

2.5. Microstructural Investigation

After the sample was etched in correct duration and correct etchant, the metallurgical phases were dissolved in different amounts from the aspect of electrochemistry, in micro scale. And the phases reflected the light characteristically due to their spectrum difference of each. We could see the microstructure of the cobalt based superalloy, which had been damaged in micro and also macro scale, by using BAB Software Optical Microscope.

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For physical structural investigation and chemical composition investigation of critical parts of micro crack, we used EDS equipped High Vacuum 20 kV scanning electron microscope which is located in Yalova University Laboratory.

2.6. Chemical Analysis and Hardness Test

For failure analysis of nozzle sample, beside the metallographic characterization, also chemical analysis and hardness tests were carried out by XRF Spectrometer, Carbon-Sulphur Analyser and Digirock HV30 Vickers Hardness Testing Machine in Gedik Holding Research and Development Laboratory and Gedik Test Center.

3. RESULTS AND DISCUSSIONS

As the first step for failure analysis of the sample which had operated for 30,000 hours in turbine engine combustion chamber, visual inspection was carried out and macro fractures and macro cracks were detected on outer surface as shown on Figure 3.

Figure 3. Failure zone detected by visual inspection

As the result of visual inspection, 2 main fracture failures and 1 macro crack failure were detected. The failure has occurred on a zone on which hot and high pressure gas might have been circulated and which is close to the connection zone with the platform. Both high temperature and pressured gas were existed around that zone and also stresses might have occurred on it. For that reason, it has been thought that the failure mechanism would be a stress-corrosion cracking.

After visual inspection, we decided to carry out optical microscopy investigations on the surface around the macro crack. Macro crack could give us some predictions about the reason of failure because it can represent the main fracture failures.

Before metallographic sample preparation and microstructure analysis processes, chemical composition analysis was carried out on the sample which had been taken far from the failure zone. According to the XRF elemental analysis result given on Table 1, it could be seen that the material is cobalt based superalloy and the chemical content values are approximate with the values of FSX 414.

Table 1. Chemical analysis result

Element	% by weight	Element	% by weight
Ni	23.8425	**Ti**	0.4329
C	0.2080	**Co**	42.0733
W	6.3959	**Cr**	22.6896
Mo	0.3453	**Si**	0.9519
Fe	0.4794	**Ta**	0.8566
Al	1.4629	**Others**	0.2617

Before the microstructural analysis, hardness tests were carried out on different points which have particular distance to macro crack center line. Hardness test results have been shown on Figure 4. Hardness values are approximate to each other in macro scale. Therefore, it was thought that locational hardening due to heat treatment effect or softening due to grain size growth should not have been occurred on the microstructure.

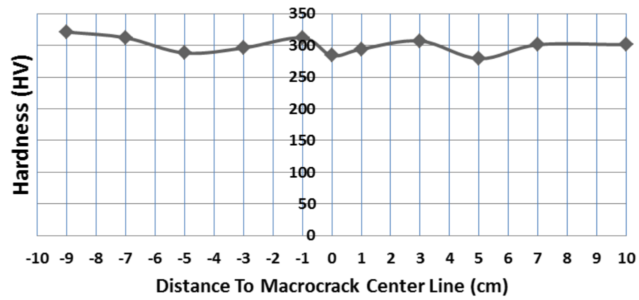

Figure 4. Hardness test result

After hardness tests, metallographic sample preparation and optical microscope investigation were carried out and as results, the microstructures below were obtained. According to these microstructures;

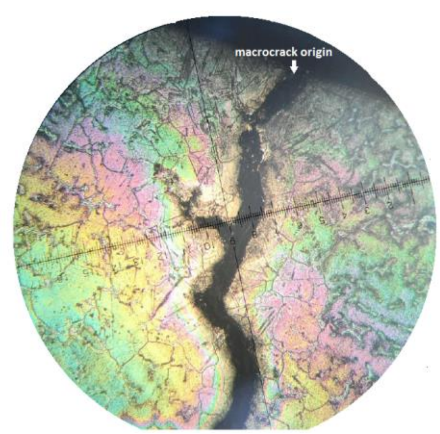

Figure 5. Microstructure of macro crack zone (100x – electrolytic etched with 6% chromic acid + 10% HCl)

On Figure 6, the microstructure of the area that macro crack had occurred and propagated through has been given. When we looked at the structure of the crack, we thought that macro crack could have occurred by growth of intergranular micro cracks. In order to understand the mechanism of failure, first, microstructural investigation of non-damaged zone was carried out. On Figure 6, the microstructure taken from zone 3 has been given.

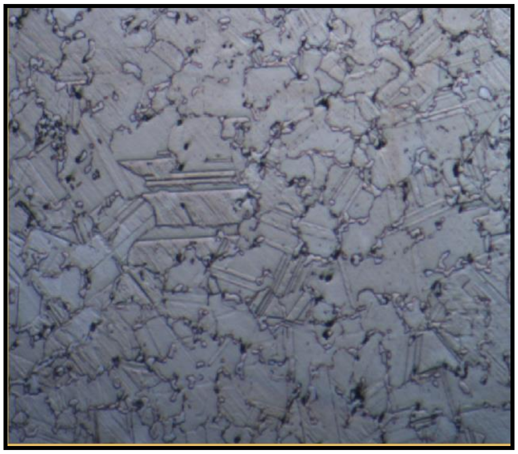

Figure 6. Microstructure of cobalt based superalloy (100x – electrolytic etched with 6% chromic acid + 10% HCl)

Twinnings inside the austenitic matrix of face centered cubic (FCC) structure and also carbide precipitations on grain boundaries and inside the grain can be seen on the microstructure that given on Figure 6. The transformation of lattice structure from face centered cubic (FCC) to hexagonal closed pack (HSP) structure occurs on cobalt based superalloys over 417^0C. In order to avoid this transition, cobalt based superalloys are alloyed with nickel [3]. Because our material includes nickel over than 10 %, it could be understood that the lattice structure of the matrix is FCC. The basic phases that affect the mechanical properties of cobalt based superalloys are the carbide phases. Therefore, the microstructures of carbides have been investigated and the results were obtained as;

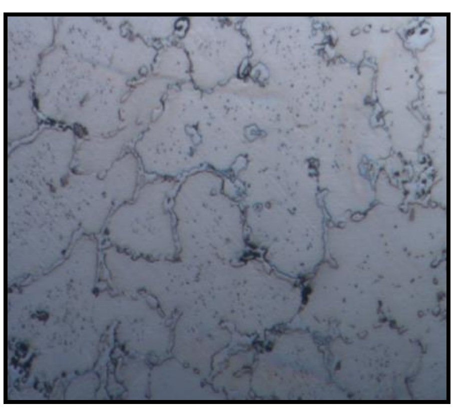

Figure 7. Microstructure of carbides obtained from non-damaged zone (100x - electrolytic etched with 6% chromic acid + 10% HCl)

The microstructure obtained from zone 2 but far from the main fractures and macro crack has been given on Figure 7. It can be seen on microstructure that spherical and chain type carbides have precipitated on the grain boundaries. However it has been detected that, the morphologies of carbides are changing by approaching to the macro crack. When the microstructure, obtained from closer point to macrocrack, given on Figure 8 is considered, it can be seen that some of the spherical and chain type carbides, are replaced by continuous formed carbides.

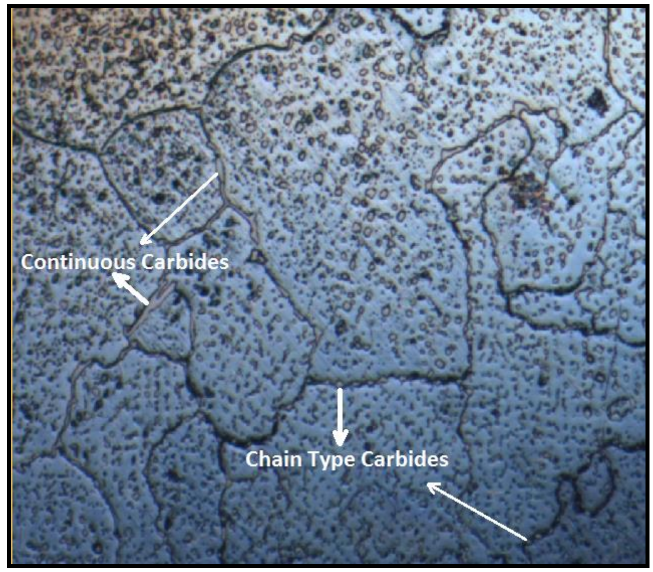

Figure 8. Microstructure of carbides obtained from closer zone to macrocrack (100x - electrolytic etched with 6% chromic acid + 10% HCl)

When we get closer to the macro crack, it can be seen that the amount of continuous formed carbides are increasing as shown on the microstructure that given on Figure 9.

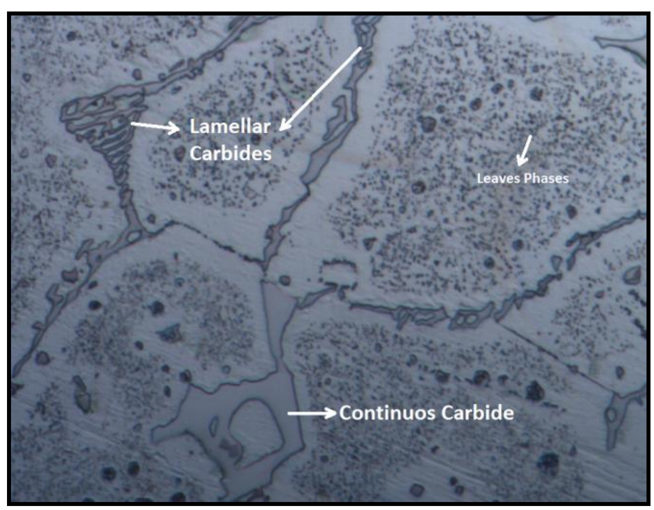

Figure 9. Microstructure of carbides near the macrocrack (200x - electrolytic etched with 6% chromic acid + 10% HCl)

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Lamellar morphology carbides and continuous formed carbides precipitated on grain boundaries and also other types of carbides and leaves phases inside the grains can be seen on the microstructure given on Figure 9.

After the detection of carbide morphology changes through the macrocrack, we decided to investigate the origin, sides and tip of the macrocrack and the microstructures are;

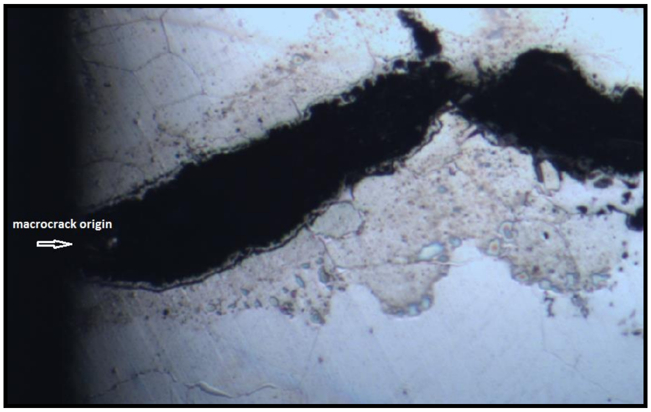

Figure 10. Microstructure of macrocrack origin (100x – slightly chemically etched with Kallings No:2)

Microstructure of macrocrack origin zone, which had been slightly chemically etched, has been given on Figure 10. Due to the fact that oxides are electrochemically affected by etchants more than metallic phases, it has been thought that the brown colored parts surrounding the crack could be hot corrosion products. After that estimation, we decided to carry out electrolytic etching.

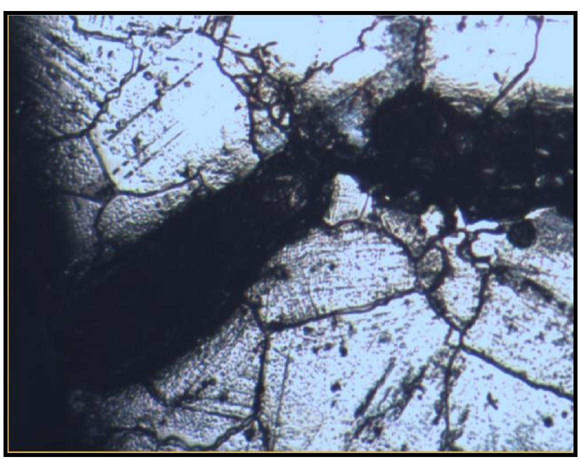

Figure 11. Microstructure of macrocrack origin (100x – electrolytic etched with 6% chromic acid + 10% HCl)

On the microstructure of macrocrack zone has been given on Figure 11, intergranular corrosion cracks and continuous carbides on grain boundaries could be seen. After that we decided to investigate the tip zone of the crack in order to see propagation mechanism of it.

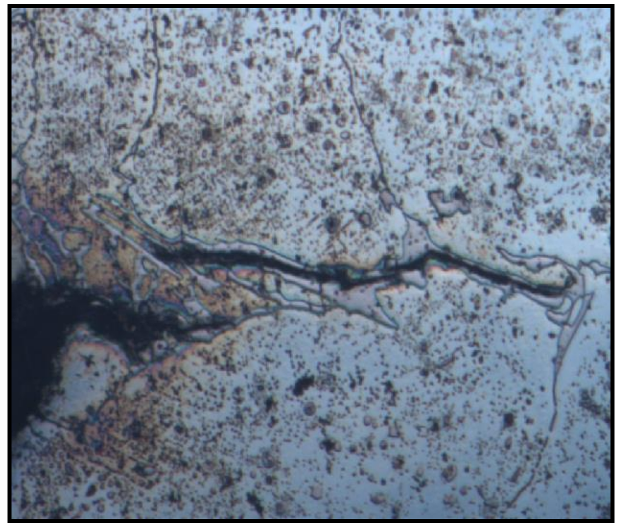

Figure 12. Microstructure of the tip zone of crack (200x slightly chemically etched with Kallings No:2)

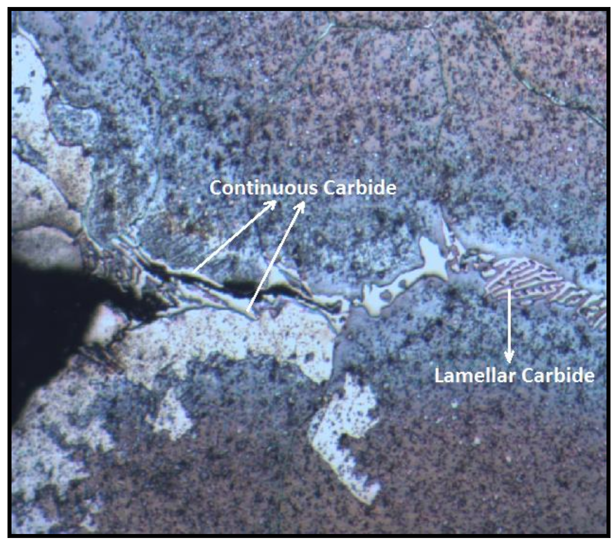

Figure 13. Microstructure of the tip zone of crack (100x - electrolytic etched with 6% chromic acid + 10% HCl)

As it can be seen on Figure 12 and Figure 13 which shows the microstructure of tip zone, macrocrack has propagated up to a point and then it continued as micro crack on its path passing through the continuous formed carbides that have precipitated on the grain boundaries as it can also be seen on Figure 14 which shows the microstructure of micro crack tip. It can also be seen that microcrack propagation stops and possibly nanocracks propagation occurs up to lamellar morphology carbides which have also precipitated on grain boundaries.

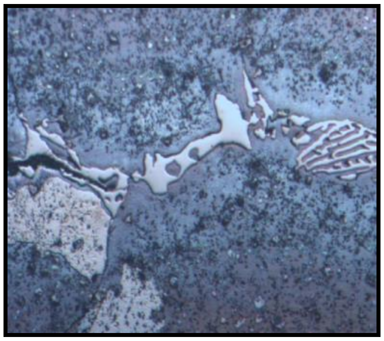

Figure 14. Microstructure of microcrack tip (200x - electrolytic etched with 6% chromic acid + 10% HCl)

After optical microscopy analysis we considered the results and decided to carry out electron microscope analysis in order to determine the types of carbides chemically and morphologically. On the Figure 15 and Figure 16, SEM images of crack zone have been shown. It can be seen that macrocrack had propagated mostly through grain boundaries zone. And also corrosion products can be seen around the macrocrack.

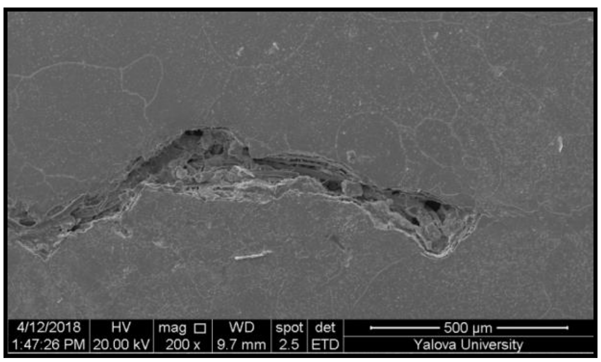

Figure 15. SEM image of macrocrack

As shown on the SEM image of macrocrack tip zone on Figure 16, macro crack propagation is blocked by semi lamellar semi continuous morphology carbide precipitates. However, macrocrack had found a path for propagation as microcrack. As it can be seen on Figure 17, the path of microcrack is surrounded by continuous morphology carbides. It means that microcrack had propagated through the continuous carbides on grain boundaries.

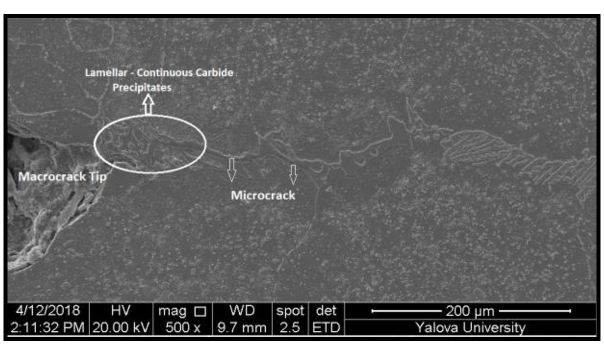

Figure 16. SEM image of macrocrack tip zone

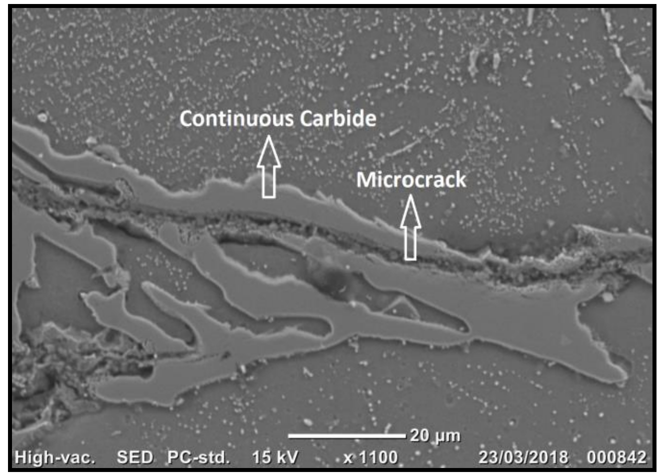

Figure 17. SEM image of microcrack

In cobalt based superalloys, $M_{23}C_6$ and M_7C_3 carbides form during the last stage of solidification and the carbide type is basically related to Cr/C ratio. While M_7C_3 carbides occur at lower Cr/C ratio, $M_{23}C_6$ carbides occur at higher Cr/C ratio [4].

SEM-EDS analysis was carried out on the points shown by blue rectangles on Figure 18. According to the results given on Table 2, continuous carbides include higher carbon and lower wolfram and molybdenum in comparison with lamellar carbides. When we compare the Cr/C ratios, while the ratio of continuous carbide is 2.012, the ratio of lamellar carbide is 2.904. As the result of SEM-EDS analysis, it could be understood that lamellar carbides are $M_{23}C_6$ and continuous carbides are M_7C_3.

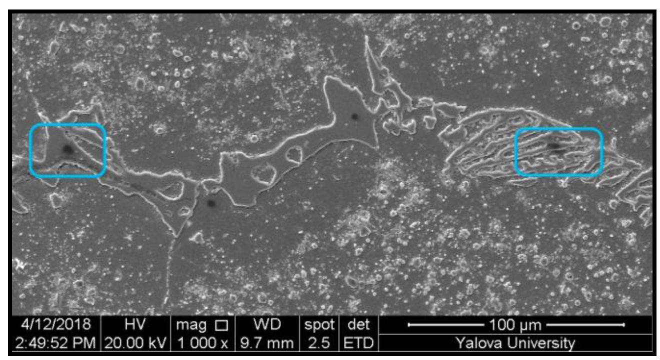

Figure 18. SEM-EDS image of carbides

Table 2. SEM-EDS analysis results

	C%	Mo%	Cr%	Co %	Ni %	Ta %	W%
Lamellar Carbide	18,31	6,97	53,18	8,97	1,48	1,57	9,52
Continuous Carbide	27,32	1,72	55,15	7,52	1,06	1,6	5,63

In super alloys, dense and continuous carbides expedite crack occurrence and propagation, decrease ductility and toughness by 30 % and shorten the lifetime of the material [5]. This could be seen on this study, too. As it can be seen on the microstructures obtained by both optical microscopy

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and electron microscopy investigations, the failure that had occurred on the nozzle, the macrocrack and microcrack, had propagated through M_7C_3 type continuous carbides and the propagation had been blocked or slowed down by $M_{23}C_6$ type lamellar carbides.

Table 3. Carbide types in cobalt based superalloys [6]

Carbide Type	Elements	Morphology
MC	Ti, Nb, Zr, Hf, Ta	Spherical, cubic or disordered shaped as particles and shinny
M_7C_3	Cr	As blocks, continuous formed and extends through grain boundaries
$M_{23}C_6$	Cr, W	Spherical, layered or lamellar morphology and on grain boundaries
M_6C	Mo, W	Randomly dispersed, pinkish white

The carbide types that occur in cobalt based superalloys are given on Table 3. The mole ratio of carbon, corresponding to 1 mole of metal, decreases when descend through the below of the table. In other words, in the presence of these elements, the amount or the possibility of formation of upper side carbides increases with the increase of carbon content.

$$Cr_{23}C_6 + 3C = 3Cr_7C_3 + 2Cr \quad (1)$$

As it can be indicated with (1), when the carbon content increases, carbide formation can occur and beside the lamellar $M_{23}C_6$ type carbides ($Cr_{23}C_6$ and $W_{23}C_6$), continuous M_7C_3 type carbides (Cr_7C_3) precipitate on grain boundaries. After that consideration, in order to see if there were any carbon content difference between the non-damaged parts and damaged part of the nozzle, we decided to carry out carbon - sulphur analysis to the sample, which has been taken from the part of the nozzle on which macrocrack had occurred. According to the C-S analysis results, strikingly, while non-damaged part of the nozzle includes 0.2 % carbon, the part of the nozzle on which failure occurred includes 0.4 % carbon. As the result of carbon analysis, it can be claimed that because of carbon diffusion into the part of the nozzle from hot gas including unburnt carbon, the carbon content of microstructure had increased and carbide transformation ($M_{23}C_6 \rightarrow M_7C_3$) had occurred.

In all super alloys, carbides are the weak phases against oxidation. In order to minimize the high temperature corrosion failure, carbides need to be small and discontinuous [7]. When all the microstructures are considered in our study, it can clearly be seen that the crack had propagated through continuous morphology carbides. In order to avoid this occurrence, the carbide transformation needs to be blocked. As a resolution advisory, molybdenum and/or wolfram contents could be increased in order to succeed it. These elements increase the potential of occurrence of M_6C carbides and M_6C carbide occurrence can decrease the potential of M_7C_3 carbide occurrence. In addition, as it can be indicated with (2), these elements increase the possibility of carbide transformation from M_7C_3 to $M_{23}C_6$. Thus, these alloying elements could help to obtain more ductile carbides on the grain boundaries and could prevent grain boundary embrittlement. So, increasing Mo and W alloying elements could increase the lifetime of a cobalt based superalloy nozzle which operates under these kinds of conditions like exposing carbon diffusion from the hot gas which includes unburnt carbon.

$$3Cr_7C_3 + 2\,(Mo,W) = Cr_{21}(W,Mo)_2C_6 + 3C \quad (2)$$

According to the carbide transformations during the service according to the (1) and (2), beside the wolfram and molybdenum content increase advisory for solution, also the content of MC type carbide making elements could be increased in order to minimize the $M_{23}C_6 \rightarrow M_7C_3$ transformation due to high chromium activity. Owing to both carbonization due to diffusion and also carbon occurrence due to transformations, instead of carbon's making brittle continuous Cr_7C_3 carbides on grain boundaries, MC type carbides such as TiC, NbC, TaC could be provided to be occurred inside the grain.

The elements, advised to be increased in cobalt based super alloy which is to be used as first stage high pressure nozzle in a jet turbine engine, in a sense, would replace with chromium and owing to this we could provide both high stress corrosion resistance and also could increase the high temperature corrosion resistance. Because in the presence of these elements, instead of brittle Cr_7C_3 carbides, more ductile $Cr_{21}(W,Mo)_2C_6$ carbides would occur on grain boundaries and also it can be provided that chromium would make Cr_2O_3 layers, which increases the corrosion resistance, instead of making carbides.

4. CONCLUSION

The cobalt based super alloy sample; which had been used as nozzle for conveying the hot and high pressure air-fuel mixture, released from combustion chamber, through first stage high pressure turbine; could have operated 30,000 hours while it had been estimated to operate 50,000-70,000 hours because of failure. In our failure analysis study, we detected the reason of the failure as carbide transformation due to carbon diffusion on to the material from circulated gas which might have included unburnt carbon. Related to the carbon diffusion, because of $M_{23}C_6 \rightarrow M_7C_3$ carbide transformation, the grain boundaries had embrittled and weakened against stress corrosion crack propagation due to the fact that continuous formed brittle M_7C_3 carbides are acting like a pathway for crack propagation while lamellar formed ductile $M_{23}C_6$ carbides are acting like a block.

We also estimated that the lifetime of the nozzle could be increased by preventing carbide transformation by increasing the molybdenum and wolfram content of the material, because they increase the possibility of $M_{23}C_6$

carbide occurrence at elevated temperatures despite carbon diffusion during service. In addition to molybdenum and wolfram content increasing advisory, we also estimated that increasing MC carbide making alloying elements content such as Ti, Nb, Ta and Hf could be beneficial for cobalt based superalloys which operate at high temperatures and under the conditions of carbon diffusion might be occurred.

Space and aerospace industry are continually developing engineering application industries. At that point, material failures should be minimized for these kinds of critical parts such jet engine parts. In order to minimize the failures, failure analysis studies should be increased and developed.

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