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Failure Analysis of Gold Dust defect in 430 grade Stainless Steel

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Abstract— Gold dust is a surface defect that usually observed on the surface of 430 grade stainless steel. Gold dusting is characterized by sparkling appearance. They occur as small flakes of metal on the cold rolled surface elongated in the rolling direction. As this defect occurs on the surface of the component, it affects the intended application of the steel for which they have been designed. Further loss of material in the powder form can affect the mechanical properties of the component. The defect is analyzed through optical and Scanning electron microscope with EDAX. From the results it is observed that the defect is due to sensitization. The paper is based on the work to identify the sources of the defect and solutions to overcome this defect.

Keywords: 430 grade stainless steel, Gold Dust, Sensitization, Surface Defect.

1. Introduction

Surface investigation of gold dust in 430 grade stainless steel involves the failure analysis of the cold rolled surface. Gold dusting occurs on the surface after the cold rolling process. The defect appears as the metal powders deposited on the on the rolled plates as shown in figure 1 that are visible to naked eye and they easily stick on the hands when toughed. These powders are usually identified by placing a transparent tape. Once the tape is removed most of the powders stick in the tape. Hence they can be easily wiped off and a simple grinding process can remove the powders on the surface. The grinding of the surface layer will not only cost a lot but also result in continuous precipitation on further ageing after treatment.

The surface grinding of rolled surface is not possible all the time. The defect is also not continuous throughout the surface of the cold rolled plate. They occur randomly on the rolled surface to certain distance. They also do not occur in all the coils of the same grade. So these are the peculiar defects that occur under certain conditions. These precipitates have sparkling appearance and the deposit appears similar to the dust on the surface and hence the name Gold dusts.

This defect is commonly called as Metal powder defect as the precipitates come from inside of the metal component. The defect usually occurs in the cold rolled surface after annealing and pickling cycle. Analysing this defect mainly requires the information regarding the condition under which the defect occurs which enables a route to find the solution.

The reason for precipitation of gold dust can be found if and only if the component which is precipitating out is identified. Thorough understanding of the cause of the failure is required to rectify this defect. The analysis of the defect is by comparison between the cold rolled plate with the gold dust defect and without the defect

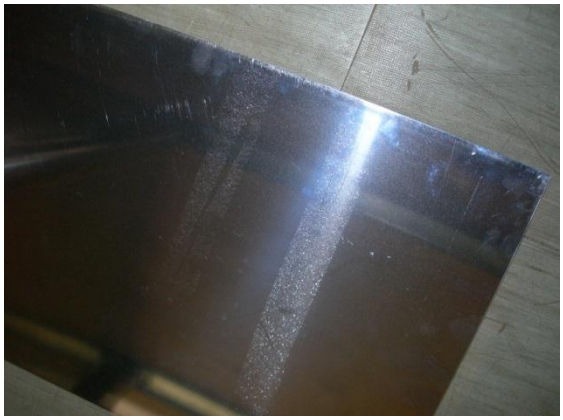

Figure 1: Gold Dust defect in 430 grade Stainless Steel

. The investigation is made through the optical microscope, Scanning Electron Microscope and Energy Dispersive Spectroscopy to compare the gold dust sample with the non-defective sample

2. Problems due to the defect

Stainless steel grade 430 is non-hardenable steel containing straight chromium, and belongs to the ferritic group of steels. This steel is known for its good corrosion resistance and formability, coupled with practical mechanical properties. It can be used in certain chemical applications due to its resistance to nitric acid. The 430 grade stainless steels have their application in Linings for dish washers, Refrigerator cabinet panels, Automotive trim, Lashing Wire, Element Supports, Stove trim rings, Fasteners, Chimney Liners where they require intact contact. The precipitation of the gold dust will disturb the lining surface. Hence the application of 430 grade stainless steel is affected. The grinding of the surface layer will not only cost a lot but also result in continuous precipitation on further ageing after treatment. The surface grinding of rolled surface is not possible all the time. Through the precipitation process one of the components of the 430 grade stainless steel is depleted. So this may affect the properties of the material under operation due to the loss of the material.

3.Experimental works:

3.1 Chemical composition of coils under examination:

A number of coils are taken of examination and the defect does not occur in the entire coil and occurs randomly at certain regions. The coils are sampled with and without defect are taken for analysis. The coil number mentioned here are for our experimental identification.The chemical composition of the coils compared is listed in the table 3.1. The samples with and without gold dust are collected from the same coil to make an effective comparison.

Table 1 Chemical composition of the coils taken for analysis

COIL NO	**C %**	**Si%**	**Mn%**	**P%**	**S%**	**Cr%**	**Ni%**	**N %**

136329	0.027	0.31	0.62	0.31	0.002	16.29	0.14	0.056
139821	0.055	0.23	0.70	0.40	0.007	16.20	0.12	0.12
139667	0.045	0.24	0.65	0.37	0.001	16.12	0.12	0.043

Several other coils are also examined to study the variation of the microstructure from case to core. The chemical compositions of the coils listed in the table are the values before rolling operation.

3.2 Microstructural analysis

The microscopic analysis is made to fine the difference between the samples with and without the gold dust at the microscopic level. As mentioned earlier once the metal powders on the surface are removed it again precipitates. This precipitation will result in the depletion of the metal which will cause the microstructure variation which can be depicted through comparison. The samples of size 4mm are cut from the rolled plate. The procedures for metallographic inspection are similar in other cases. As they are stain less steels electrolytic etching is done

Electrolytic etching: Specimen should be connected to terminal of the unit and stainless

steel sheet of approximately 50 X 30 mm size to be attached to negative terminal. For 430 grade Ferritic stainless steels, Etchant is 5%HCl +95% Methyl alcohol, Current density: 3.8 Amp/cm^2, Voltage: 5 V, Time of Etching: 30 sec.

The micrographs with and without gold dust defect for two different coils are shown in the figure 2 and 3.

Coil no.136329

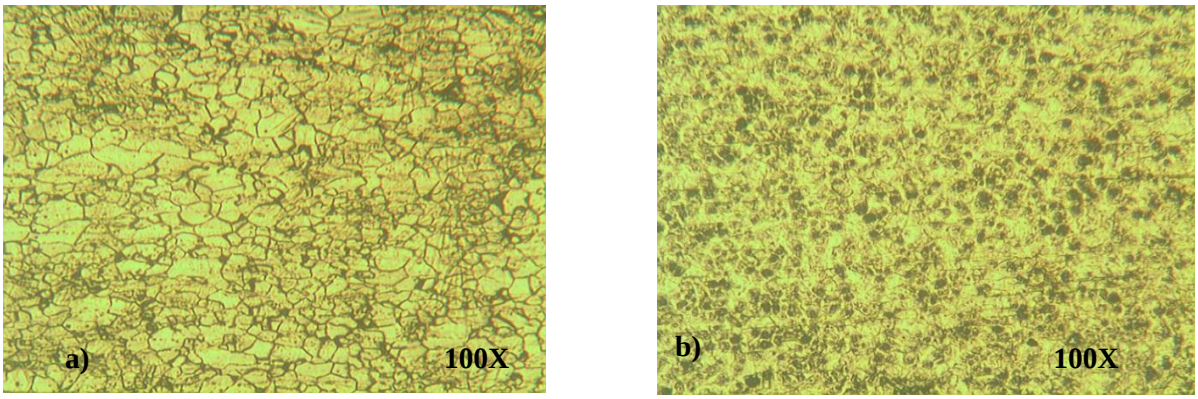

Figure 2: a)Fine recrystallized structure with distributed carbides (Micrograph of defect free sample) b)Partially recrystallized structure with carbide precipitates at grain boundaries (Micrograph of defect sample).

Coil no.139821

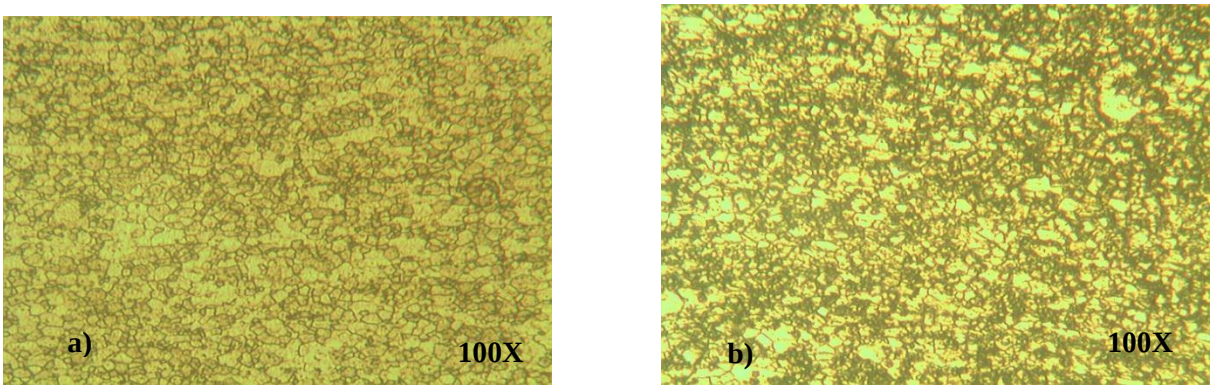

Figure 3: a) Fine recrystallized ferritic grains of size 8 throughout the structure **(**Micrograph of defect free sample). b) Fine recrystallized ferritic grains of size 8 throughout with profuse carbide precipitation. **(**Micrograph of defect sample).

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The sample with the defect shows profuse precipitation when compared with the defect free sample. The comparison of the microstructure from case to core is also made which is shown in the figure 4. The grain boundaries are broken in the case whereas towards the core they are complete.

Coil No: 139667

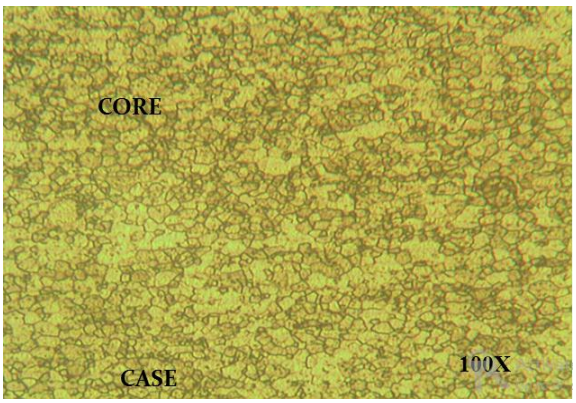

Figure 4 : Comparison of Case and Core.

3.3 SEM Analysis

To view the gold dust at higher magnification and for the better topographical analysis Scanning Electron Microscope is used. The specimen preparation is required before SEM analysis. The samples of thin strips of rolled plate of 25 X 25 mm and 0.8 mm thickness were taken for analysis. SEM images were taken till 1000X for comparison.

The SEM image of the defect free sample at 1500X magnification is shown in the figure 5(a). The SEM image of gold dust sample at 150X is shown in figure 5 (b). The SEM image of gold dust sample at 1000X is shown in figure 6.

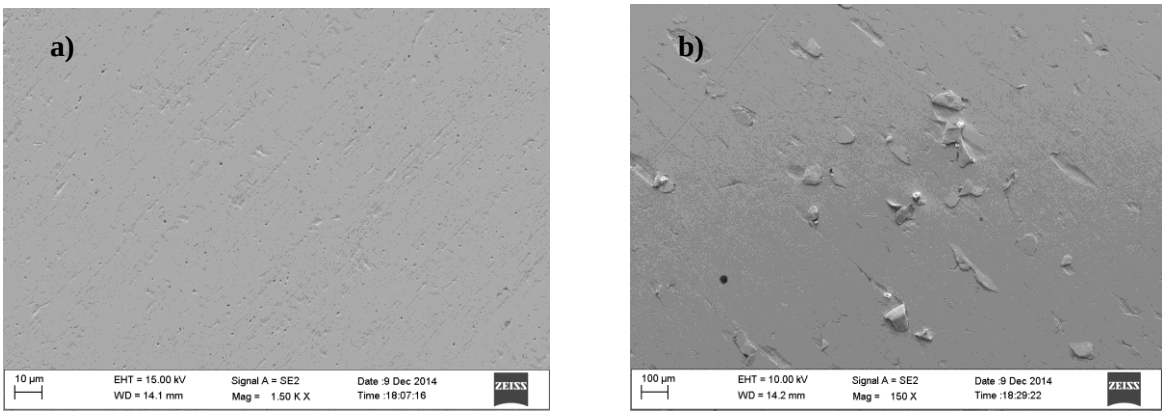

Figure 5: a) Scanning elecron photomicrograph of cold rolled surface of 430 grade stainless steel at 1500X Coil no.139821 (without gold dust defect). b) Scanning elecron photomicrograph of cold rolled surface of 430 grade stainless steel at 150X Coil no.136329 (with gold dust defect).

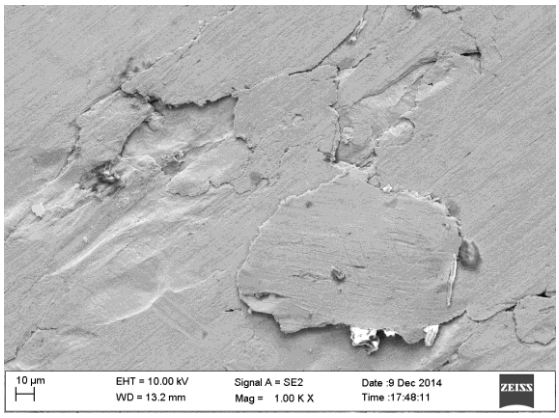

Figure 6: Scanning elecron photomicrograph of cold rolled surface of 430 grade stainless steel at 1000X , Coil no.136329 (with gold dust defect).

3.4 EDAX Analysis

Energy dispersive X-ray spectroscopy (EDS) is a technique to detect characteristic X-rays on the basis of their energy to obtain chemical composition of a specimen bombarded by a focused beam of electrons. The EDAX is employed to know the chemical composition of the metal powder. Thus the material which precipitates out can be analyzed through the EDAX image.

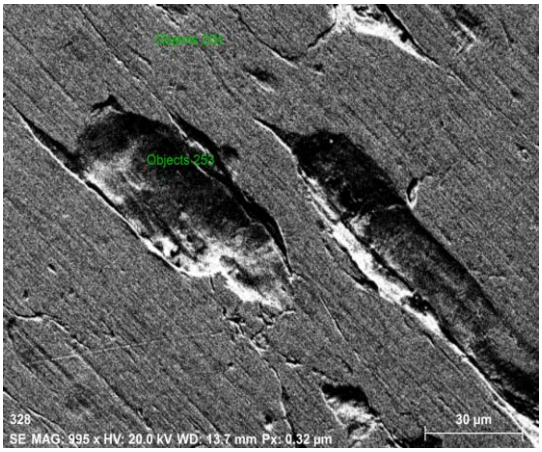

Figure 7: Micrograph from energy dispersive spectroscopy.Analysis is done at two points: one at the defect area and another on the suface near the defect.

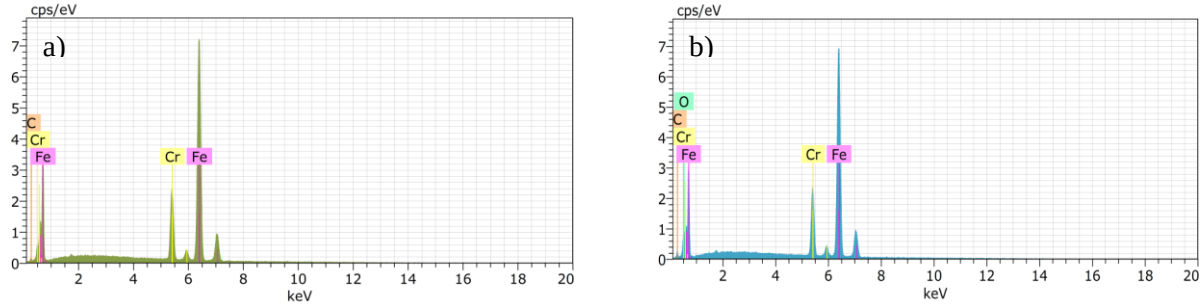

Figure 8: a) Graph obtained at the area of the defect at point 254 in Energy Dispersive Spectrometer micrograph b) Graph obtained at the area near the defect at point 253 in Energy Dispersive Spectrometer micrograph

Table 2 The Spectrum obtained from objects 254 shows

El	AN	Series	unn. C [wt.%]	norm.C [wt.%]	Atom.C [wt.%]	Error (1 sigma)
Fe	26	K series	80.25	82.68	73.59	2.17
Cr	24	K series	13.81	14.22	13.60	0.41
C	6	K series	3.01	3.10	12.81	0.76
		Total	97.07	100.00	100.00	

Table 3: The Spectrum obtained from objects 253 shows

El	AN	Series	unn. C [wt.%]	norm.C [wt.%]	Atom.C [wt.%]	Error (1 sigma)
Fe	26	K series	78.93	78.87	61.48	2.13
Cr	24	K series	12.87	12.86	10.77	0.38
C	6	K series	5.83	5.82	21.11	1.19
O	8	K series	2.44	2.44	6.64	0.49
		Total	100.08	100.00	100.00	

The chemical composition of the specimen at two points are taken one on the surface of the gold dust and the other on the matrix near the defect. The variation in the chemical composition is shown in the graph and the spectrum at two points such as spectrum 254 and 253 are listed below the graph in figure 8 a) ad b) respectively.

The EDAX results show that there is a significant decrease in the chromium percentage and increase in the carbon percent and oxidation has occurred at or near the region of the defect when compared with the matrix far away from the defect.

4. Observation and Inference

From metallurgical micrograph (Figure 2(a) and Figure 3(a)), there is fine recrystallized grain grains whereas in in Gold dust sample shown in figure 2(b) and 3(b), the areas near the grain boundaries appear black indicating that there is a profuse carbide precipitation at the grain boundaries as a result of sensitization. Further from the figure 4 the grain boundaries are distorted towards the surface due to the precipitation.

In Scanning Electron Microscope and Energy Dispersive Spectroscopy, Gold dust samples show a significant decrease in Cr% and increase in C% clearly indicating that the area affected by defect suffers Chromium depletion and the related Chromium Carbide precipitation. There is also increase in O_2% which is due to the fact that there is no sufficient Cr for passivation and hence an oxide formation has occurred at the surface.

5. Conclusion

From the results of chemical analysis, microstructure and SEM analysis we find that this defect is accompanied by a Chromium Carbide precipitation. Visual observation shows that this defect is characterized by a sparkling appearance, which results from small flakes of metal on the cold rolled surface. The apparent cause of such defect is nothing but “The sensitization of ferritic stainless steel which occurs during the annealing process”.

Use of a low carbon alloy will completely eradicate the above problem but while considering the compatibility of such steel with respect to the application it is required, there are a number of practical ways to prevent or lower the sensitization which include:

1. Limiting of the annealing temperature to 840°C (which is still sufficient for recrystallization).

2. Lowering of the cooling rate after annealing (by the use of batch annealing, for example), and a limiting of grain growth.

3. Stabilization using Titanium or Niobium-which readily forms carbides than chromium and hence lowering the chances for Chromium Carbide precipitation.

6. References

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