Sensitization of Gold Dust in 430 Grade Stainless Steel

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Abstract— *'Gold dusting' is a surface defect that is sometimes observed on cold-rolled AISI type 430 stainless steel. Gold dusting is characterized by a sparkling appearance, which results from small flakes of metal on the cold rolled surface, the flakes are mostly elongated in the rolling direction. The processing steps include continuous casting, hot rolling, continuous annealing, and pickling. One possibility is that the flakes of metal are grains that had been undercut by intergranular corrosion such intergranular corrosion may occur during pickling after the annealing step (which itself follows hot rolling). If intergranular corrosion does occur during this pickling step, the intergranular cavities would be elongated by subsequent cold rolling; this can account for the observed morphology of gold dusting. If the steel has been sensitized, intergranular corrosion may occur during pickling. Pickling is commonly carried out by electrolytic descaling in a neutral sodium sulphate solution followed by immersion in a nitric acid/hydrofluoric acid bath. The sensitized type 430 stainless steel does, indeed, suffer intergranular corrosion in a nitric acid/hydrofluoric acid bath, while it is largely unaffected during electrolytic pickling. Hence, sensitization is a possible cause of gold dusting.*

Keywords— ***430 grade stainless steel, Gold dusting, Intergranular corrosion, Pickling, Sensitization.***

I. INTRODUCTION

Surface investigation of gold dust in 430 grade stainless steel involves the failure analysis of the cold rolled surface. 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. This chapter deals with the investigation of defect under optical microscope, Scanning Electron Microscope and through that causes of the defect are studied and based on that causes suggestions for rectification can be provided.

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.

Grade 430F stainless steel is usually provided in bar form to be used in automatic screw machines. Grade 434 has similar properties as grade 430, although it is a molybdenum-bearing version. The molybdenum content enhances its corrosion resistance.

1.1 Difficulties in Processing 430 Grade

Ferritic stainless steels get corroded in chloride and sulphurdioxide containing industrial and marine atmospheres the 17% Cr-ferritic steels are less corrosion resistant than austenitic stainless steels in reducing atmospheres. As no phase change occurs on heating in fully ferritic stainless steels, grain refinement is difficult. Grain growth is rapid on heating in such a single phase microstructure as diffusion too is faster in BCC structure. Ferritic stainless steels start coarsening rapidly at 600^0C compared to 900^0C for austenitic stainless steels. Thus, after a high temperature such as welding, the grains become very coarse and are difficult to be refined. It is a major drawback.

As ferritic stainless steels have BCC structure, they show ductile to brittle transition, and this impact transition temperature is considerably higher than that for mild steel due to embrittlement effect of chromium dissolved in ferrite. The 25% chromium steel will be brittle even at room temperature if the carbon content exceeds 0.03%. As ferrite grains cannot be refined, the presence of coarse grains further raises the ductile to brittle transition temperature.

As ferritic stainless steels have BCC structure, these steels have lower general ductility and higher yield strength than austenitic stainless steels. The ferritic steels are inferior where stretch forming is required such as in deep drawing for applications such as kitchen sinks, domestic catering utensils, although ferritic steels can be readily coined, embossed, cold forged, or spinning can be done easily. Ferritic stainless steels show stretcher strains during drawing or stretching.

A more serious problem common in ferritic stainless steels is 'ridging, or roping', which is not due to yield-point phenomenon but to a crystallographic textural effect. Rope marks are depressions on one side of the sheet, matching an elevation on the other side; the thickness remains constant and the markings are parallel to the rolling direction. These mar the appearance of parts formed from sheet.

II. PROBLEMS DUE TO THE DEFECT

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 component 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.

III. EXPERIMENTAL WORK

3.1 Microstructure Examination

All samples irrespective of grade to be cut and mount in a fashion such that the plane of polish is perpendicular to the forming (rolling) direction. We can perform hand grinding with a simple device in which four belts of abrasive paper (240-, 320-, 400- and 600-grit). Running water is supplied to cool specimen surfaces during hand grinding. Grinding produces damage that must be minimized by subsequent grinding with finer abrasives. In particular, two things must be followed to ensure optimal results. First, specimens are rinsed with running water to remove surface debris before switching grinding belts; and second, specimens are rotated 90◦ from the previous orientation. Abrasives for polishing are usually diamond paste, alumina or other metal oxide slurries. Polishing includes coarse and fine polishing Electrolytic etching

Etching is done to etch the polished surface of the specimen using electrolytic etching unit for stainless steel and dip etching for carbon steels.

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: 5%HCl +95% Methyl alcohol, Current density: 3.8 Amp/cm^2, Voltage: 5 V, Time of Etching: 30 sec. The microstructures obtained through optical microscope are

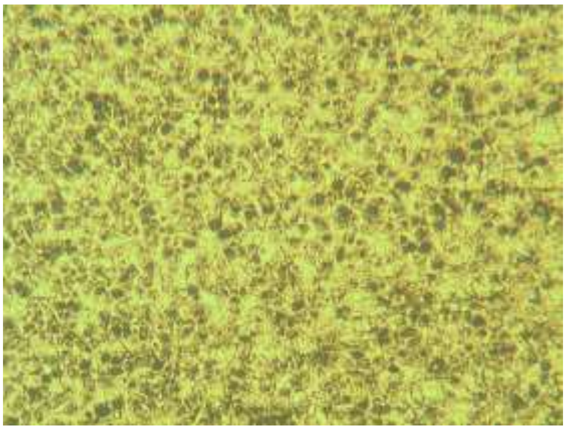

Fig.3.1.1: Partially recrystallized structure with carbide precipitates at grain boundaries.

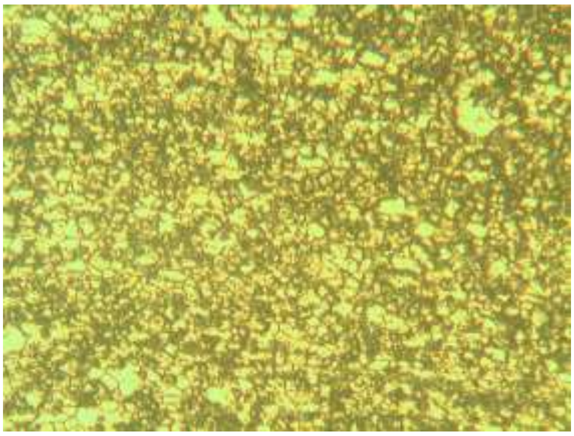

Fig.3.1.2: Fine recrystallized ferritic grains of size 8 throughout with profuse carbide precipitatation.

3.2 SEM Analysis

SEM has a large depth of field, enabling characterization of fractured surfaces. The combination of high magnification, larger depth of focus, greater resolution and ease of specimen observation. SEM is the one of the most used instruments for material characterization. The SEM images (Fig 3.2.1and Fig 3.2.2) reveal that gold dust formed comes from material not from the external atmospheres during their manufacturing process .Thus the material had undergone chemical reaction during the transformation.

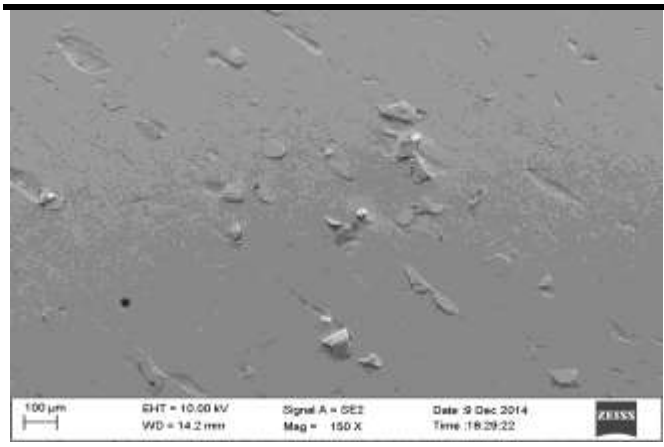

Fig.3.2.1: Scanning elecron photomicrograph of cold rolled surface of 430 grade stainless steel at 150X

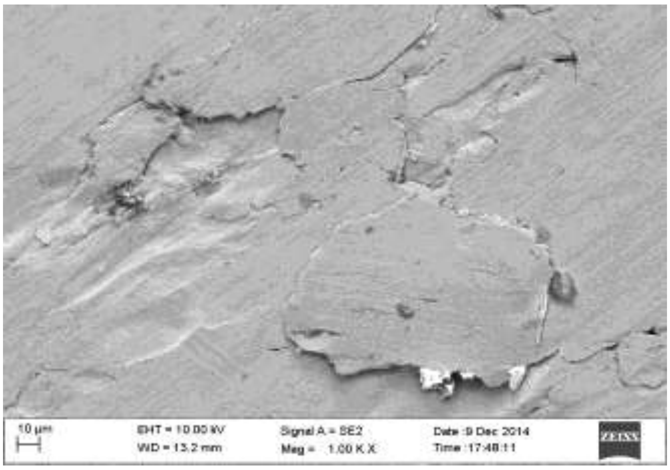

Fig.3.2.2: Scanning elecron photomicrograph of cold rolled surface of 430 grade stainless steel at 1000X

The product formed as a result of reaction can be analysed through energy dispersive spectroscopy

3.3 EDAX Analysis

Energy dispersive X-ray spectroscopy (EDS) is a technique to detect characteristic X-rays on the basis of their energy rather than wavelength to obtain chemical composition of a specimen bombarded by a focused beam of electrons

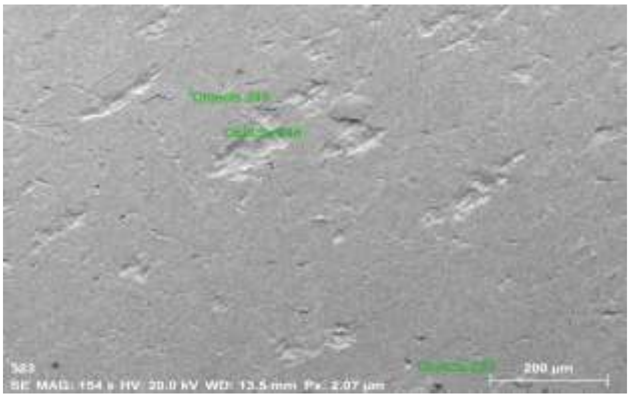

Fig 3.3.1: Micrograph from energy dispersive spectroscopy.Analysis is done at two points: one at the defect area and another on the suface near the defect

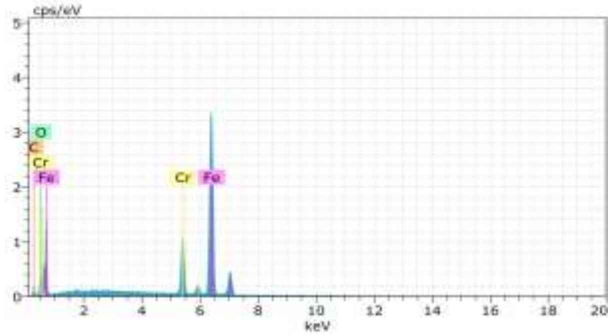

Fig.3.3.2: Graph obtained at the area of the defect at point 248 in energy dispersive spectrometer micrograph

The Spectrum obtained from objects 248 shows

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
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	

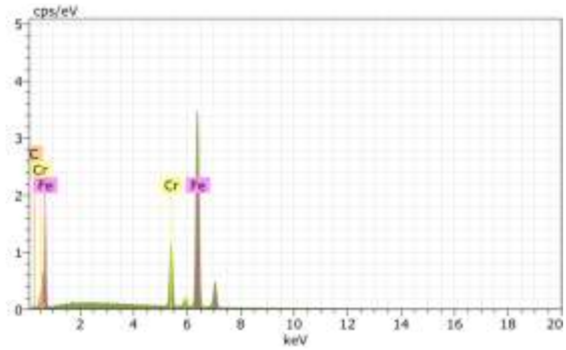

Fig 3.3.3: Graph obtained at the area near the defect at point 249 in energy dispersive spectrometer micrograph

The Spectrum obtained from objects 249 shows

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
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	

IV. OBSERVATION AND INFERENCE

From metallurgical micrograph (Fig 3.1.1 and Fig 3.1.2), in Gold dust sample, the areas near the grain boundaries appear black indicating that they are empty, the grain boundaries are distorted towards the surface. 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. The increase in % O_2 is also due to the above fact that there is no sufficient Cr for passivation and hence an oxide formation at the surface.

V. 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”.

VI. RECOMMENDATIONS

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.

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