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Measuring Carbon in Steel Using Calibration Curves on the Microprobe; Failed Cap Screw Study

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A fractured cap screw was presented for failure analysis to determine the cause of the failure. The broken screw and a matching "sister" screw from the same flange have anomalously high hardness values,52.6 HRC, Spec= 38-45HRC.

Hardness measurement is a straight forward technique to estimate strength.

Question is whether the anomalous high hardness came from the steel chemistry, a heat treatment problem, or some other source.

Figure 1.
Broken cap screw and sister screw

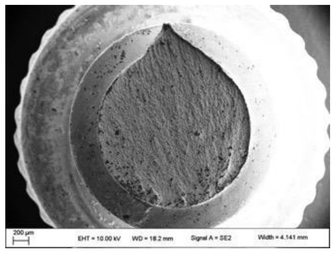

Figure 2. SEM Micrograph of screw head side of the fracture surface.

A Calibration Curve approach was used on the microprobe as Carbon content was targeted as the most likely cause of failure.

Carbon content is a controlling variable in steel mechanical properties.
As carbon weight % increases the steel strength and hardness also increase.
However, the steels ductility and toughness decrease with increasing carbon weight %.

Thus, it was important to the resolution of this failure analysis to have independent measures of carbon content in the screws.

A JEOL 8530F EPMA was used to measure Carbon content of the cap screw.
Analysis Conditions were 15keV, 40nA, with a 20um probe diameter. Measured C counts are shown in Table 1.
A calibration curve was established from five steels with a range of known carbon, Fig. 3

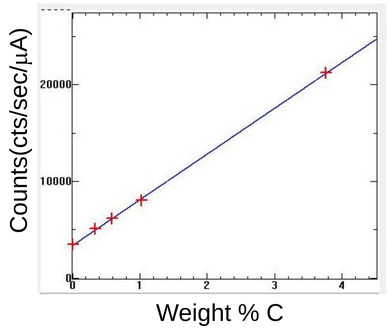

Figure 3. Carbon standards calibration curve data

Point	Cts/sec/µA
1	8977.5
2	9250.8
3	9418.9
4	9667.8
5	9272.1
6	8718.8
7	8784.0
8	8435.0
9	9960.9
10	8756.3
11	9463.3
12	10091.7
13	8909.1
14	9830.8
15	9251.5
16	9555.1
17	8825.8
18	9049.6
19	8781.2
20	8717.9

Table 1. Broken cap screw C Counts

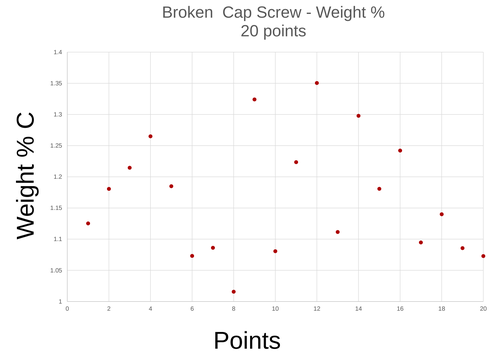

Figure 4. Broken cap screw carbon weight %, based on Carbon calibration Curve for 20 points

Using the calibration curve method, the carbon content of the failed screw was determined to be 1.2 weight % with a S.D. % of 0.1. The specified composition is 0.28-0.33 weight %.

LECO Gas Combustion Analysis was used to determine the carbon composition of another suspect screw, which was 1.0 weight %.This result is in excellent agreement with the calibration curve method used in the EPMA measurements.

The screw fracture is attributed to the screw being produced from an incorrect alloy. The alloy composition is consistent with SAE-AISI 52100 Bearing Steel, a high carbon, high hardness alloy not generally suited for tensile loading, i.e. threaded fasteners, due to the low fracture toughness of this alloy.

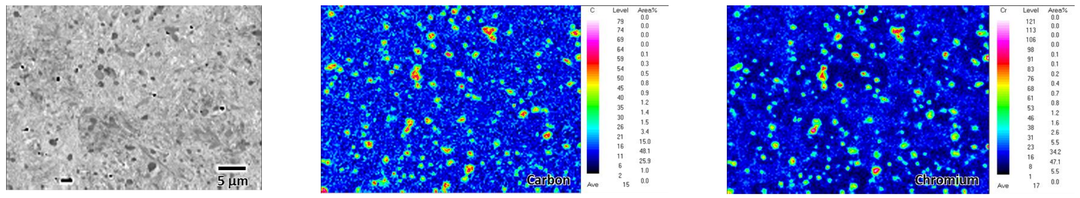

Figure 5. BSE image and WDS maps of C and Cr showing spatial distribution of carbides on fractured cap screw

References:
[1] T. Yamashito et al., Scientific Reports, Nature.com, DOI:10.1038/srep29825.
[2] G. V. T. Ranzetta and V. D. Scott, J. Sci. Instrum, 43(1966), p. 816

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