CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 58-216

INVESTIGATION OF BRITTLE FRACTURES OF CHROMIUM PLATED TENSILE TEST BARS OF SAE 4340

by

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Copy No. 8

DECEMBER 12, 1958

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SUMMARY OF RESULTS

Both ductile (40% Reduction of Area) and brittle (0% Reduction of Area) test bars were examined. No metallurgical nor significant chemical deficiencies could be detected. Most important, examination showed no perceptible difference between ductile and brittle bars. By the process of elimination, it was concluded that hydrogen embrittlement was the cause of the lack of ductility. The reasons for the variation in hydrogen embrittlement lie in the processing.

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(3 tables, 7 illus.)

INTRODUCTION

On 19 June, 1958, Mr. A. Sankoff of Canadian Vickers Limited, Montreal, Que., submitted halves of two chromium plated tensile test bars (Figure 1) which had fractured with little or no reduction of area with a request for an opinion as to the reason for the lack of ductility. A copy of their test report was appended. The relevant data from this report are shown in Table 1.

Table 1

Extract from "Report of Physical Tests"

Sample Designation	Ultimate Tensile Strength	% Reduction of Area
1 I.D.	230,000	Nil
1 O.D.	275,000	Nil
2 I.D.	276,500	40.7
2 O.D.	271,000	40.2
3 I.D.	267,500	37.6
3 O.D.	285,500	38.9

The material is SAE 4340, heat treated to a nominal tensile strength of 260,000 to 280,000 psi, with a specified minimum reduction of area of 30.0%. While the heat treatment applied to the samples was not given, the specified heat treatment calls for austenitizing at 1525 - 1575°F, oil quench, double temper within one hour at a temperature dependent on the as-quenched hardness and varying from 400 - 500°F. Also electroplated parts must be baked within one hour at a temperature of 370°F for a period of 8 hours. The material is finally given a retained austenite stabilization treatment consisting of holding at 250°F for 24 hours. At our request, halves of the ductile tensile bars (Nos. 2 and 3) were forwarded and were received on 8

July, 1958.

VISUAL EXAMINATION

Visual examination of the "brittle" bars showed the fracture to be symmetrical about a longitudinal plane in both cases. Figure 2 shows how the flat central portion of the fracture extends to the surface in one area only and is surrounded by a shear lip around the remainder of the circumference. Striations on the flat portion of the fracture indicate the fracture origin to be at the surface (Figure 3a). No such striations were observed on the flat portions of the ductile fractures (Figure 3b). This, of course, is consistent with the normal case where the tensile fracture originates at the centre of the section. The extensive "craze" cracks observed on the surface of the bars are normal for hard chromium plate. There was no evidence of oxidation in any portion of the fracture surfaces.

CHEMICAL ANALYSIS

Drillings were obtained from four of the samples for chemical analysis. Due to limitation of sample size, only the more important elements shown below were determined.

Table 2

Chemical Analysis

	Percentage of Element				
	Carbon	Manganese	Nickel	Chromium	Molybdenum
1 I.D. (Brittle)	0.44	0.69	1.78	0.64	0.37
2 I.D. (Ductile)	0.44	0.67	1.77	0.64	0.36
1 O.D. (Brittle)	0.44	0.69	1.78	0.65	0.37
2 O.D. (Ductile)	0.48	0.69	1.76	0.63	0.36
SAE 4340	0.38/0.43	0.60/0.80	1.65/2.00	0.70/0.90	0.20/0.30

It is apparent from the above that the samples do not quite conform to the specified analysis, being somewhat on the high side in carbon and molybdenum and low in chromium. However, the discrepancy in carbon is covered by the permissible tolerance on check analysis (except for 2 O.D.). It is not considered that the discrepancies in the contents of chromium and molybdenum are significant. On the contrary, the uniformity of composition in the face of the variation in ductility is most significant.

MICROEXAMINATION

Longitudinal sections through the fractures and through the bases were prepared from samples 1 I.D., 1 O.D., 2 I.D. and 2 O.D. A considerable number of cracks normal to the surface were noted in the area of the fracture on 1 I.D. (Fig. 4). Two similar cracks were noted on 1 O.D. and none on 2 I.D. or 2 O.D. There was no evidence of oxidation nor of chromium plate in the cracks in the steel. From this, it is inferred that the cracks were formed during tensile testing. There did not appear to be any significant difference between the four samples in the roughness of the steel surface. Some slight ductility was noted immediately adjacent to the fracture of 1 O.D. There was no perceptible evidence of ductility in the fracture

of 1 I.D. The chromium plate of 1 O.D. was more extensively cracked than that of 1 I.D. This may well be due in part to the fact that 1 O.D. did yield slightly prior to fracture, whereas 1 I.D. did not.

In the unetched condition, all samples showed about the same non-metallic inclusion count and distribution. Samples 1 I.D. and 2 I.D. were etched with ethereal picric + 4% zephiran chloride and no evidence of temper embrittlement was detected. Etching with picral showed no difference between the brittle and ductile samples. Etching with 2% nital showed the microstructure of all samples to consist principally of tempered martensite with some bainite (Figure 6). Again, there was no perceptible difference between the brittle and the ductile material. As a final check on the comparative microstructures, carbon extraction films were examined with an electron microscope. As Figure 7 shows, the carbides in the martensite plates are similar. In fact, the carbide configuration is typical for a low temperature (about 400°F) temper subsequent to quenching to martensite.

HARDNESS TESTING

Vickers hardness tests using a 50 Kg load were made on transverse sections through the bases of four samples. The results, shown in Table 3, are uniform.

Table 3

Vickers Hardness Test Results

Sample	Hardness	
	Vickers	R_c (Converted)
1 I.D.	565	53
1 O.D.	549	52
2 I.D.	544	52
2 O.D.	546	52

A transverse microhardness traverse, using a 500 gram weight and a Knoop diamond indenter, was run adjacent to the fracture from the circumference to a depth exceeding the depth of the shear lip on sample 1 O.D. The hardness varied only from R_c53 to R_c54 (Converted from Knoop Hardness Numbers). This indicates the shear lip is not due to a soft ductile surface layer.

DISCUSSION

As has been brought out previously, no compositional or structural defects were observed in any sample. More important, no significant differences of chemistry or microstructure could be detected between the brittle and the ductile bars.

Two possibilities suggest themselves as reasons for the brittleness. The less likely of the two is misalignment in the tensile testing machine. The fracture origin being at one side rather than in the centre of the section can be construed as indicating such a cause, particularly in view of the high strength level of the steel. However, in view of the rather considerable ductility indicated by the reduction in area at the fracture of the other samples, it is considered highly improbable that misalignment in itself and with no embrittlement of the material, can be held responsible.

The second possibility is hydrogen embrittlement. This is, of course, an ever-present danger with pickling and electroplating processes and measures are taken to guard against it. While the usual measures are generally sufficient to relieve obvious hydrogen embrittlement in medium strength steel (200,000 psi or less), they may or may not be sufficient in the case of high strength steels. This variability in results follows from the extreme sensitivity of the

ultra high strength steels and the usual variability of the many factors operative. This sensitivity can be illustrated by the results of experiments in which tensile test bars of SAE 4340 were heat treated to various strength levels and then were electrolytically charged with hydrogen for various times. The degree of embrittlement was shown by the drop in reduction of area. The results showed that at the minimum charging time of 2 minutes the 200,000 psi bars still exhibited full ductility whereas the 270,000 psi bars had dropped from an uncharged value of 44% reduction of area to only 4%; i.e., the ductility was reduced by a factor of 1 for the 200,000 psi bars and a factor of 10 for the 270,000 psi material. As a final illustration of the extreme sensitivity of very high strength steel, it is pointed out that laboratory investigations of the problem commonly encounter abnormal scatter of results despite elaborate precautions to maintain controlled conditions. The foregoing has been introduced to emphasize the point that very subtle differences in processing conditions and material can lead to very pronounced differences in mechanical properties in the ultra high strength steels and, as a corrollary, control measures that are perfectly satisfactory for lower strength material will not necessarily suffice for this material.

The above indicates the strong probability of hydrogen embrittlement being the reason for the lack of ductility encountered. It remains to account for the fracture characteristics noted in terms of hydrogen embrittlement. The two outstanding characteristics of the fractures were the location of the fracture origin (at the surface instead of at the centre of the section) with the accompanying indication of brittleness at this point, and the presence of a ductile shear lip adjacent to the remainder of the circumference. To deal with

the second feature first, a ductile shear lip would be expected at a free surface in the presence of partial but not extreme embrittlement. The situation in these bars after fracture had commenced is analogous to that in a plate fracturing at a temperature somewhat below its transition temperature. Under these conditions, the crack front forms a roughly elliptical outline with the locus at the centre of the section. The fracture is brittle in the interior of the plate where the triaxial restraint is high but becomes ductile and forms a shear lip at the surface (side of the plate) where the triaxial restraint decreases as the free surface is approached. It was demonstrated that the shear lip was not due to a soft ductile surface layer. The presence of the shear lip further demonstrates that the surface layer is not appreciably more brittle than the interior of the bar; i.e. diffusion has occurred permitting the original surface concentration to be dissipated to give a relatively uniform distribution of hydrogen throughout the bar. Due to the mobility of hydrogen this can occur easily and relatively quickly ($\frac{1}{2}$ hour at 300°F would probably be sufficient).

Qualitatively, the surface fracture origin can be accounted for in terms of notch sensitivity. The high strength steels are, of course, highly notch sensitive. Embrittlement, from whatever cause, would be expected to enhance this notch sensitivity and, further, to do so in proportion to the tensile strength of the steel. The cracks in the chromium plate would act as notches, initially at least. Then too, the steel surface is not perfect and there is always the possibility of an inclusion at the surface. The fact that no evidence of a prior notch could be detected subsequent to fracture does not, of

course, even suggest let alone prove that a small notch was not in fact there.

CONCLUSIONS

(1) No significant metallurgical or compositional deficiencies were detected.

(2) No difference could be detected between brittle and ductile bars.

(3) By a process of elimination, it is concluded that hydrogen embrittlement is the most probable reason for the lack of ductility.

(4) The reasons for the apparent variation in hydrogen embrittlement are considered due to variations in the processing and are beyond the scope of this investigation.

DRB/RB

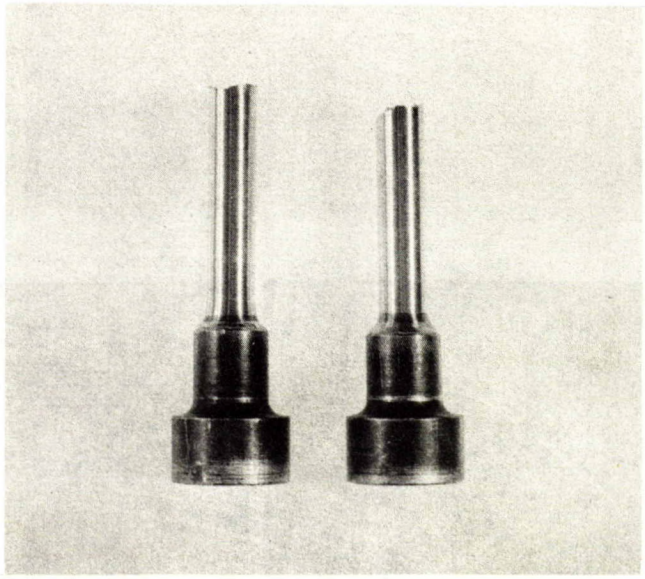

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Fig. 1. - Brittle fractures, No. 1 I.D. on the left, and No. 1 O.D. on the right.

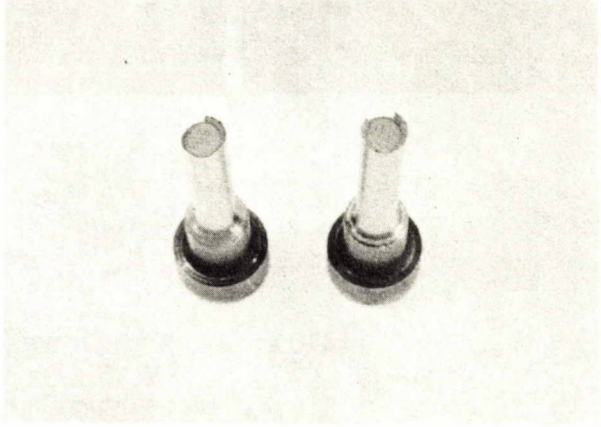
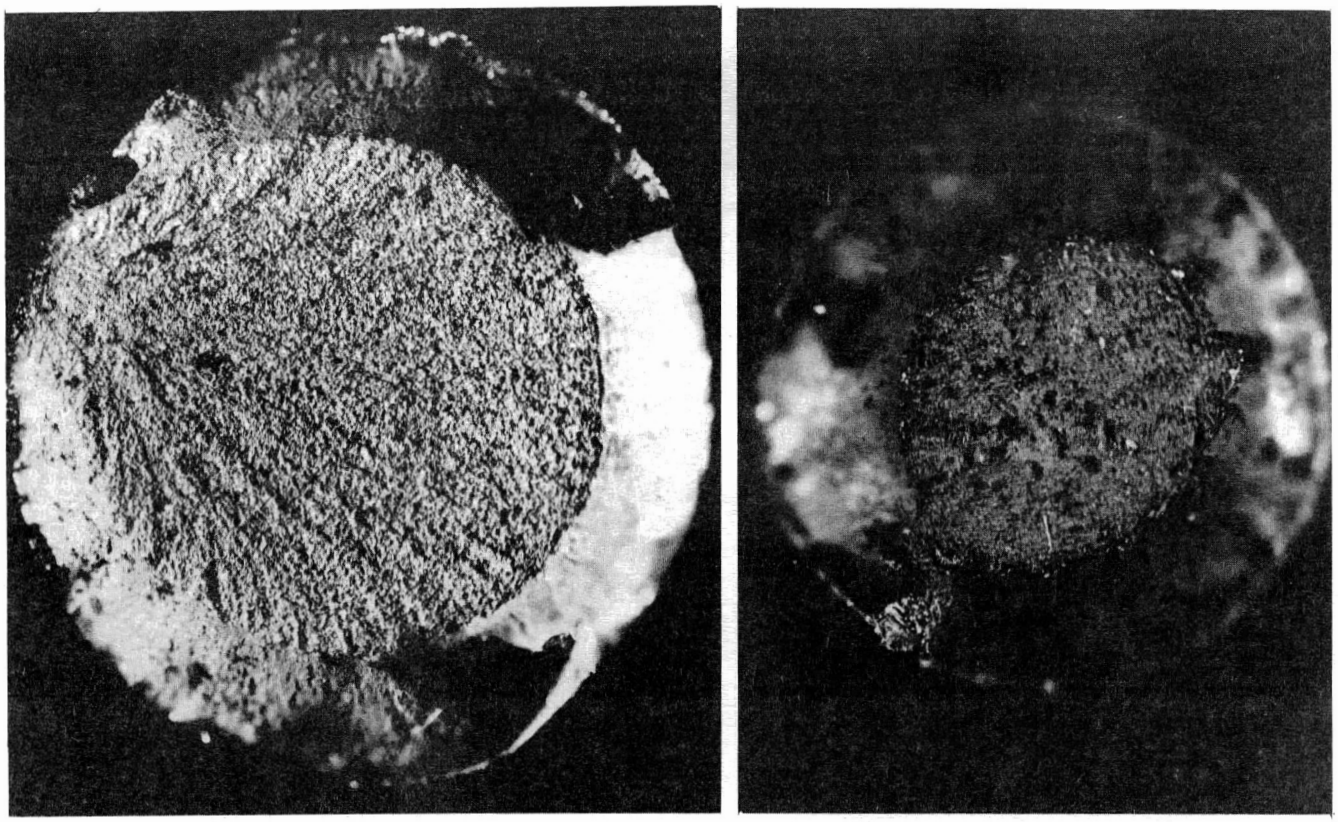


Fig. 2. - Brittle tensile bars as above. Note the symmetry about a longitudinal plane with the central plane extending to the circumference in one area, and a shear lip around the remainder of the circumference.

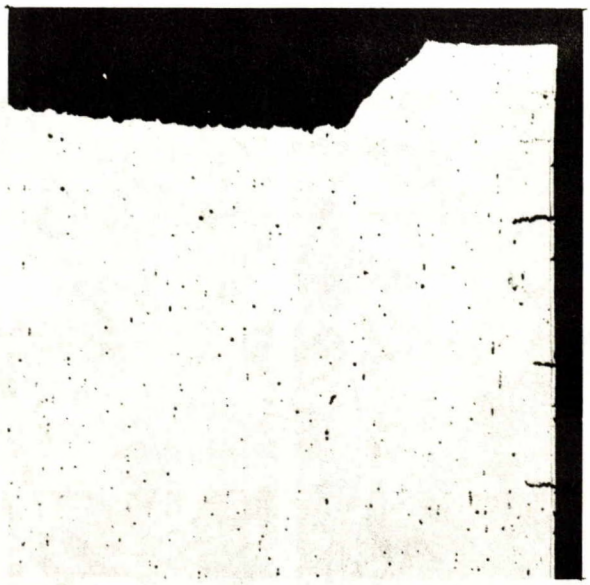


(a) (b)

(X10)

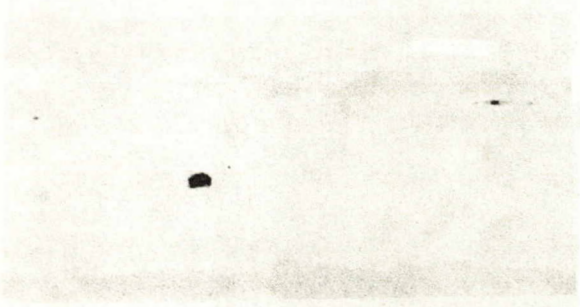
Fig. 3 (a). - Brittle fracture of 1 O.D. Note striations indicating the fracture origin to be at the surface at the left.

(b). - Ductile fracture of 2 O.D. Note lack of striations.



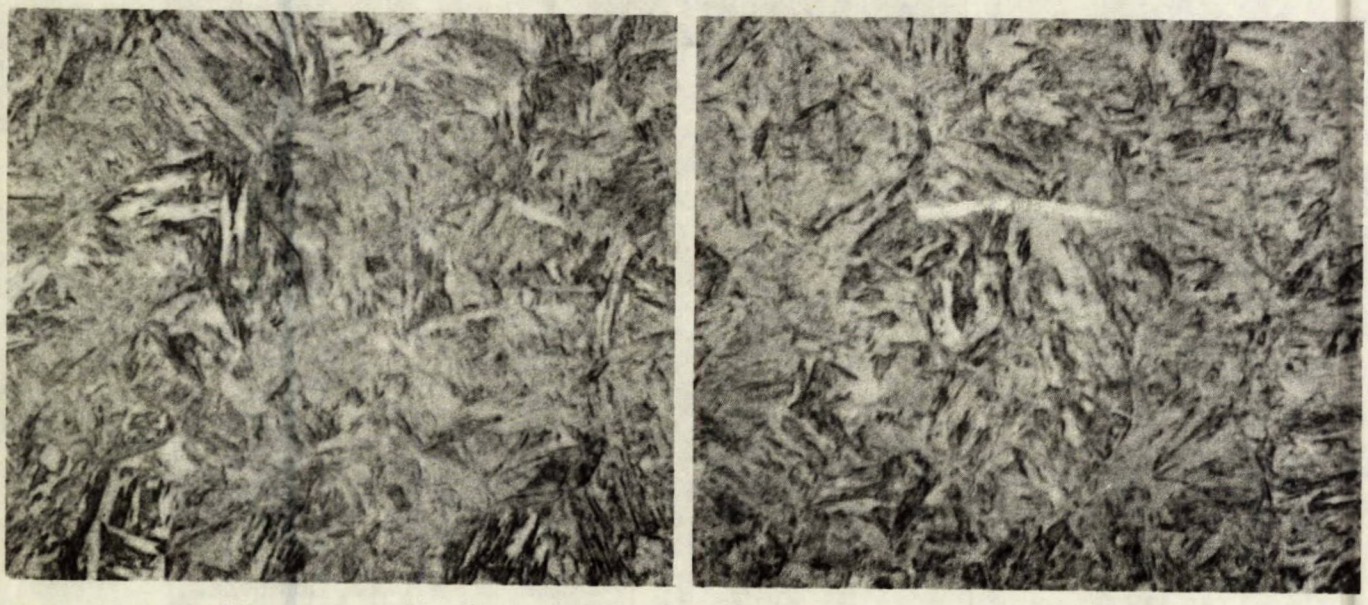
(X16, unetched)

Fig. 4. - Longitudinal section through the fracture of 1 I.D. Note the cracks normal to the surface at the right.



(a) (X100, Unetched) (b)
Sample 1 I.D. Sample 2 I.D.

Fig. 5. - Note similarity in inclusion count and distribution and the comparative cleanliness of the steel in both samples.

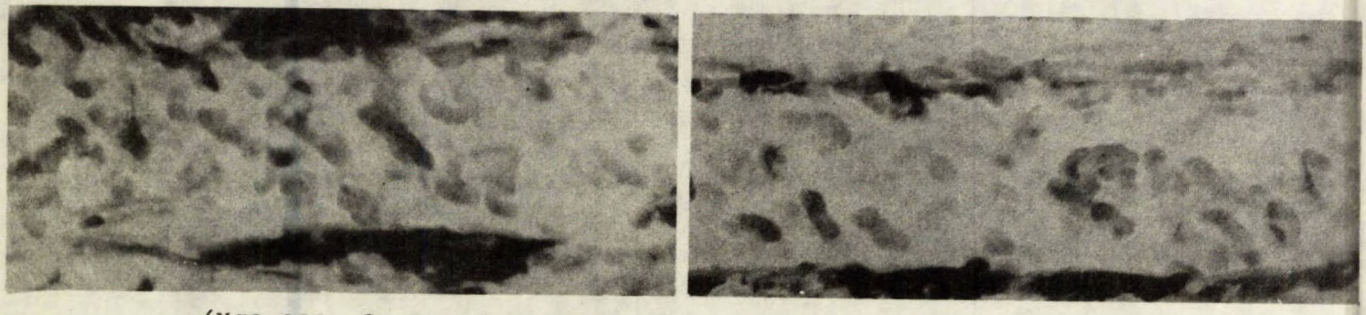


(X1500; 2% Nital Etch)

(a) (b)

Sample 1 I.D. Sample 2 I.D.

Fig. 6. - Microstructures typical of quenched and tempered material, consisting principally of tempered martensite with some bainite. Note there is no apparent difference between the brittle and the ductile material.



(X50,000; Carbon Extraction, Electron Micrograph; 2% Nital Etch)

(a) (b)

Sample 1 I.D. Sample 2 I.D.

Fig. 7. - Note similarity of configuration of the carbides in the martensite plates.