This document was produced by scanning the original publication.

Ce document est le produit d'une numérisation par balayage de la publication originale.

DECLASSIFIED
DATE OCT 22 1979
INDUSTRIAL CONFIDENTIAL
AUTHORIZED BY W.H. Erickson

IR 65-24

CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 65-24

METALLURGICAL EXAMINATION OF THREE BROKEN LEAF SPRINGS

FOR REFERENCE

NOT TO BE TAKEN FROM THIS ROOM

by

D.A. MUNRO & D.E. PARSONS

PHYSICAL METALLURGY DIVISION

COPY NO. 14

MARCH 25, 1965

Declassified
Déclassifié
Industrial Confidential

Mines Branch Investigation Report IR 65-24

METALLURGICAL EXAMINATION OF THREE BROKEN LEAF SPRINGS

by

D. A. Munro* and D. E. Parsons**

- - - - - - - - - - - - - -

SUMMARY OF RESULTS

Examination of three broken leaf springs manufactured from AISI-6150 steel showed that the vanadium content of the steel was 0.22% and that the steel had not been aluminum-killed.

Evidence of numerous slag inclusions and of microsegregation indicated some unusual factor in teeming.

The presence of a banded microstructure suggested that the heat treatment given this steel had involved use of a direct quench from a high austenitizing temperature. The high austenitizing temperature was indicated by a relatively coarse grain size.

The springs were used in service at Rockwell 'C' 47, corresponding to the optimum tempering temperature for AISI 6150 steel springs. There was evidence of precipitated vanadium compound outlining primary austenite grains, probably due to slow cooling, after forging, in the interval between forging and liquid quenching.

Recommendations were made concerning aluminum deoxidation and maximum spring-forming temperature.

*Technician and **Senior Scientific Officer, Ferrous Metals Section, Physical Metallurgy Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

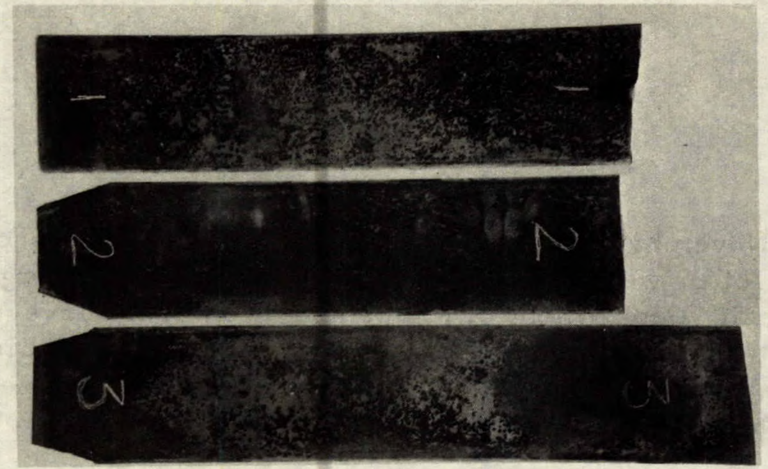
INTRODUCTION

On November 12, 1964, three failed leaf spring sections with a covering letter were submitted to the Physical Metallurgy Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, by Mr. R. Watson, Chief Metallurgist of Western Steel Limited for metallurgical examination to determine any possible cause of premature failure of these springs.

VISUAL EXAMINATION

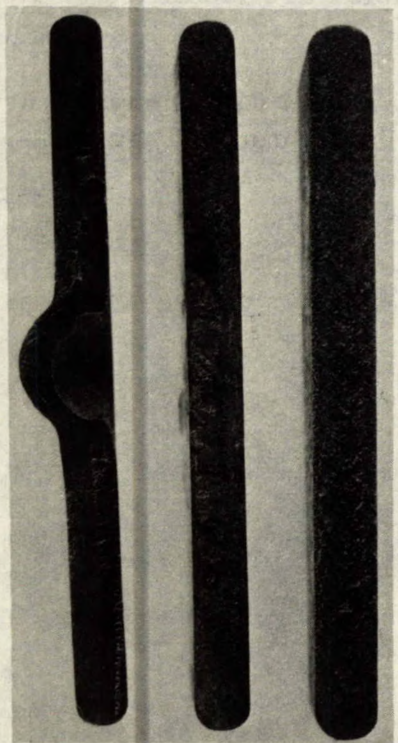
The appearance of the as-received spring sections is illustrated in Figure 1. The samples are identified as No. 1, 2 and 3 for purposes of this report.

The fracture surfaces were heavily rusted; however, these were cleaned and are illustrated in Figure 2.



X1/5 Actual size

Figure 1. Broken Leaf Springs, No. 1, 2, 3 - Fractures visible at right of picture.



1 2 3
Actual size

Figure 2. Fracture Surfaces, Samples No. 1, 2 and 3. The fractures appear to be low-cycle, high-stress, fatigue fractures with cracks initiating at both edge surfaces and propagating inwards to meet at the centre. The thickness of the three spring leaves was 0.28 in., 0.30 in., and 0.34 in., No. 1, 2 and 3, respectively.

CHEMICAL AND SPECTROGRAPHIC ANALYSES

The results of chemical analyses, made on drillings obtained after sand-blasting, and of semi-quantitative spectrographic analyses are shown in Table 1.

TABLE 1

Chemical Composition (Per Cent)

Element	Spring No. 1	Spring No. 2	Spring No. 3	AISI 6150H	AISI 6152
Carbon	0.55	0.55	0.50	0.48/0.53	0.48/0.55
Manganese	0.85	0.85	0.75	0.60/1.00	0.70/0.90
Silicon	0.40	0.40	0.38	0.20/0.35	0.20/0.35
Sulphur	0.017	0.013	0.019		
Phosphorous	0.021	0.021	0.012		
Chromium	1.15	1.15	1.20	0.75/1.20	0.80/1.10
Vanadium	0.22	0.22	0.22	0.15 min.	0.10 min.
Molybdenum	0.03	0.03	0.06		
Nickel	0.05	0.05	0.11		
Copper	0.19	0.19	0.28		
Aluminum*	<0.01	<0.01	<0.01		
Arsenic^Φ	ND	ND	ND		
Antimony^Φ	ND	ND	ND		
Tin^Φ	0.007	0.01	0.009		

* Acid soluble aluminum was not detected by wet analytical methods. (The composition of the springs is close to maximum limits with respect to carbon, silicon, chromium and contains a relatively large quantity of vanadium).

Φ Semi-quantitative spectrographic results are shown for As, Sb and Sn. Other determinations are appended.

HARDNESS SURVEY

Hardness traverses were made on transverse sections from each spring using a Tukon hardness tester and 500 g load. The results of these tests, Table 2, show that samples No. 1, 2 and 3 have average hardnesses of Rockwell 'C' 46, 47 and 48, respectively. The springs were each decarburized for a depth of 0.006 in., in the sections tested. (The hardness, R_C 47, corresponds to a tempering temperature of approximately 800°F and provides an optimum hardness-impact condition for spring leafs.)

TABLE 2

Distance from Surface, in.	Hardness (Tukon Survey, Transverse Sections, - 500 g Load)		
Spring No. 1	Knoop Hardness	Rockwell 'C', Converted	Remarks
0.002	376	38	Decarburization
0.004	465	45	0.006 in. depth
0.006	465	45	
0.008	509	46	- Average
0.010	509	46	hardness=R_C 46
0.020	509	46	
0.030	509	46	
0.040	509	46	
0.050	509	46	
Spring No. 2			
0.002	423	42	Decarburization
0.004	486	46	0.005 in depth
0.006	502	47	
0.008	533	47	- Average
0.010	533	47	hardness=R_C 47
0.020	533	47	
0.030	533	47	
0.040	533	47	
0.050	533	47	
Spring No. 3			
0.002	400	40	Decarburization
0.004	483	45	0.006 in depth
0.006	483	45	
0.008	538	48	- Average
0.010	538	48	hardness=R_C 48
0.020	538	48	
0.030	538	48	
0.040	538	48	
0.050	538	48	

METALLOGRAPHIC EXAMINATION

Longitudinal sections from each leaf spring were examined. Large stringer inclusions were observed in samples No. 1 and 3. Sample No. 2 also contained numerous stringer inclusions somewhat smaller and shorter than those of samples No. 1 and 3. Etched longitudinal sections are illustrated in Figures 3, 4 and 5. Banding is apparent in all the as-received, quenched and tempered specimens.

Figures 6, 7 and 8 illustrate the appearance of longitudinal sections etched in saturated aqueous picric acid-tridecylbenzene sulfonate solution. This etch has clearly delineated prior austenite grain boundaries in a manner which suggests that the metal may be embrittled.

Similar areas are shown, etched in the same manner, after portions of each spring were reheat treated* by normalizing from 1650°F, reheating to 1550°F, oil quenching and tempering at 700°F (R_C 51), followed by water quenching from the tempering temperature. The grain boundary constituent has been eliminated, as illustrated in Figures 9, 10 and 11.

Reheat treatment by normalizing, austenitizing, oil quenching, tempering and water quenching was also effective in producing a much finer grain size. Broken Charpy V-notch bars after reheat treatment had a Shepherd** fracture size of No. 8, whereas the Shepherd fracture number was 5 in the as-received condition.

---

* Retempering as-received metal for 1 hr at 700°F or 1050°F, followed by water quenching, did not dissolve the grain boundary constituent nor eliminate banding.

** As illustrated, p. 413, Metals Handbook published by the American Society for Metals, 1948 edition.

IMPACT TESTS

Comparison of 0.197 in. (half size) Charpy V-notch impact bars in the as-received and in the reheat treated (normalized, austenitized, oil quenched, tempered, water quenched) conditions was made using seven bars from each group. All bars were broken at 75°F. In the presence of the V-notch, results for both groups of substandard bars were similar, and gave relatively low values of 2 to 4 ft-lb. The V-notch was standard and was located on the 0.197 in. side. The tests were repeated using unnotched bars, struck in the same manner as the notched bars in an attempt to demonstrate the notch effect of the grain boundary constituent present in the austenite grain boundaries. The results are shown in Table 3.

TABLE 3

Impact Tests Using 0.197 x 0.394 x 2.165 in. Bars
Room Temperature

Test Condition		Charpy V-Notch on 0.197 in. Side	Unnotched
As-received	(a)	4	146
" "	(b)	4	150
" "	(c)	4	142
" "	(d)	4	130
" "	(e)	3	136
" "	(f)	3	140 Av. 141 ft-lb
Reheat-treated	(a)	2	152
Normalized 1650°F	(b)	3	190
Austenitized 1550°F O.Q.	(c)	2	170
Tempered 700°F W.Q.	(d)	3	170
" " " "	(e)	2	160
" " " "	(f)	3	166 Av. 168 ft-lb

The impact results on half size notched bars in both the as-received and reheat condition suggest that at spring hardness the material is brittle in the presence of a V-notch. However, the results on unnotched bars show some increase in energy required for fracture, 168 ft-lb vs. 141 ft-lb.

The increased toughness of unnotched bars after reheat treatment is attributed to elimination of grain boundary constituent present in the as-received samples and to refinement of grain size.

The tempering temperature selected (700°F) for the laboratory reheat treatment tests corresponded to the minimum point for AISI 6150 steel as illustrated in Table 4. Higher values would have been obtained by use of an 800°F tempering temperature corresponding to the production heat treatment (R_C 47).

TABLE 4

Data* for 1/2 in. Diameter AISI-6152, Quenched and Tempered as Shown

Tempering Temperature	Hardness R_C	Izod Impact Value
600°F	53	11 ft-lb
700°F	50	9 ft-lb
800°F	46	14 ft-lb

* Properties of Carbon and Alloy Steels - published by Bethlehem Steel Company.

HEAT TREATMENT TESTS

Duplicate samples from springs No. 1, 2 and 3 were reheat-treated by normalizing (1650°F); austenitizing (1550°F); oil quenching and tempering at 700°F. After tempering, one set was water quenched, the other set was air quenched. Banding and grain boundary constituent were eliminated in both sets.

Samples in the as-received condition were retempered at 700°F and were water-quenched, air quenched, or furnace cooled after tempering. Retempering at 700°F did not eliminate banding nor dissolve the grain boundary constituent.

Similar retempering of as-received material at 1050°F did not effect any major change in banding or grain boundary constituent, except that additional precipitation occurred within the grains when the samples were slow cooled, in air, or in the furnace from 1050°F.

A final series of heat treatment tests was done in which metal was first heat treated by normalizing, austenitizing, oil quenching and tempering to dissolve the grain boundary constituent, then was reheated to 1950°F (forging temperature) and was slow cooled (50°F/hr) to 1600°F followed by water quenching, tempering and metallographic examination. Slow cooling from 1950°F to 1600°F resulted in development of a heavy, continuous film completely outlining the grain boundaries. Several quench cracks were observed and these followed the brittle grain boundary constituent as illustrated in Figure 13.

DISCUSSION AND SUMMARY OF OBSERVATIONS

The vanadium contents of AISI-6150 and AISI-6152 steels are specified as 0.15% minimum and 0.10% minimum, respectively. The springs examined contained averaged contents of 0.22% V with spectrographic indication of higher than this average quantity in segregated areas. Special steel mill and heat treatment precautions to avoid slow cooling at forging and rolling temperatures are required when vanadium contents exceed about 0.15%.

The analytical results also show that the acid soluble aluminum content of the samples was at the trace level, signifying that the steel had been deoxidized by silicon-vanadium practice rather than by the more conventional silicon-aluminum fine grain practice. Data pertaining to other alloy steels in the 0.40-0.50% carbon range and at spring hardness show a very considerable improvement in the impact-transition curve when aluminum deoxidation is used at the optimum level. (Acid soluble aluminum residual content, approximately 0.02-0.03%). Probably AISI-6150 steel of competitive manufacture contains less vanadium (close to the specified minimums 0.10-0.15%) and has been deoxidized with an optimum quantity of aluminum.

Aluminum deoxidation, coupled with restriction of the vanadium content, would facilitate forging and heat treatment by the spring manufacturer with respect to grain coarsening and precipitation of embrittling grain boundary constituent if springs are slow cooled from the forging temperature (about 1800°F) to a lower temperature (about 1550°F) followed by direct quenching and tempering.

As demonstrated by heat treatment tests, salvage of existing material may be possible by renormalizing and rehardening to effect solution of grain boundary constituent and grain refinement, providing quench cracks are not present.

The steel samples examined were relatively dirty, containing numerous and long slag laminations. However, examination of the fractures showed no definite relation between inclusion content and failure.

Very pronounced banding was observed in the three failed, as-received springs, probably indicating slow cooling in the interval between forging and completion of the martensitic transformation. Reheat treatment of the three springs by normalizing, austenitizing and tempering followed by air cooling masked the banding (revealed by etching with saturated aqueous picric acid and tridecylbenzene sulfonate) - and dissolved the grain boundary constituent. The original segregation is revealed as banding whenever the steel is slow cooled and is traceable to ingot casting conditions. (Homogenization of the structure, at temperatures above 1950°F was not attempted; however, published data show that temperatures of the order of 2300°F are necessary to effect homogenization of severely segregated vanadium steel—hence, the best control of coarse ingot structure is at the steel mill by control of pouring temperature and ingot freezing characteristic.)

In addition to composition and steel mill variables, the Shepherd grain size of the as-received springs was relatively coarse, Shepherd No. 5, whereas after reheat treatment there was no evidence of banding (effectively masked) or of grain boundary constituent, and the Shepherd grain size was much reduced, - to Shepherd No. 8.

It was also observed that all three of the "as-received" springs responded to the aqueous picric acid + trideclybenzene sulfonate etch by showing strong attack at primary austenite grain boundaries. (This response is stated to be indicative of temper embrittlement.) This grain boundary response was not eliminated by simply retempering and water quenching — however, it was eliminated by normalizing, austenitizing, oil quenching, tempering followed by water quenching.

SUMMARY

In summary, it is believed that the competitor's AISI 6150 steel would be aluminum-killed and would contain the minimum quantity of vanadium (0.10% to 0.15% max). In this condition it would be less susceptible to precipitation of vanadium compound during slow cooling after forging and prior to direct quenching— it would also tend to remain fine grained at forging temperature and would not be liable to quench cracking. The forging temperature should also be controlled to avoid grain coarsening prior to hardening.

Despite the maximum 0.35% Si allowed by specification, usual wrought spring steel practice is to adhere to the range 0.15% to 0.25%.

From the viewpoint of the spring manufacturer, the embrittling vanadium compound can be eliminated by resort to reheat treatment involving normalizing, hardening and tempering, followed by water-quenching. (This constitutes two additional heat treatments that may possibly be warranted from the viewpoint of quality, but are probably unnecessary with the competitor's material on account of the presence of 0.02% - 0.03% residual aluminum coupled with a considerably lower vanadium content).

It should also be noted that Western's AISI 6150 steel samples contain considerably more numerous and longer slag inclusions than is normally observed, and that the ingot segregation in this chromium-vanadium steel was evidenced by the appearance of banded microstructure in the quenched and tempered as-received spring samples.

Use of the Western AISI 6150 steel may be possible, providing slow cooling and coarse grain size are avoided, particularly after forging, and if the high inclusion content can be tolerated. (There is no evidence in the fractures observed to prove that the impact-fatigue life of the three springs was directly affected by the inclusion content). Slack-quenching should be avoided in the presence of 0.22% V.

The success of the forging and heat-treating operation could be confirmed on trial springs by use of the aqueous picric acid-tridecylbenzene sulfonate etch to ascertain that banding and grain boundary precipitation are not present. (Improvement of the inclusion situation should be attempted in future melts).

e.g. For Trial Heat Treatment:

- Forge
- Reheat to 1650°F* - air cool rapidly or oil quench
- Austenitize at 1550°F - oil quench
- Temper at 775°-800°F for 1 hr - water quench.

* The normalizing treatment effects grain refinement but does introduce surface decarburization. The decarburization can be minimized and compensated for by shot peening. Alternatively, it may be possible to oil-quench from 1650°F.

CONCLUSIONS

1. The springs failed by high-stress, low-cycle fatigue (impact fatigue).

2. The vanadium content, 0.22%, was higher than the specified minimum. Similar steel of competitive manufacture tends to the minimum specified vanadium value. The Western steel was silicon-vanadium deoxidized and did not contain the usual residual, acid-soluble, aluminum content 0.02-0.03%. The silicon content was higher than is usual in this grade of steel 0.40% vs. 0.15-0.25%.

3. The steel contained more numerous and larger stringer inclusions than are usual in spring steel; however, nothing was observed to suggest that premature failure was directly related to the high inclusion content.

4. The steel was strongly banded suggesting that:-

   (i) the steel mill ingot was segregated; this effect was not eliminated by rolling.

   (ii) after forging at the spring plant, the springs cooled slowly to the hardening (direct, liquid quenching) temperature.

5. The microstructure of the leaf springs and the response of the steel to saturated aqueous picric acid + wetting agent suggests that they are embrittled by precipitation of a vanadium compound in primary austenite grain boundaries. This phase probably precipitated (in this susceptible steel) when springs were allowed to cool from forging temperature to the temperature required for hardening by direct liquid quenching. The relatively coarse grain size observed in broken Charpy bars is consistent with a direct quench heat treatment after forging.

6. The fracture grain size of as-received material was Shepherd size No. 5. After reheat treatment by normalizing, austenitizing, oil quenching and tempering the steel grain size was refined to Shepherd size No. 8 and the grain boundary constituent was dissolved. (Grain coarsening occurred during forming of spring samples 1, 2 and 3.)

7. Metal from the springs was reheat treated to dissolve the grain boundary constituent, then was heated to 2000°F, held for 1/2 hr, slow cooled at 50°F per hr to 1600°F, followed by quenching. This heat treatment developed a continuous brittle film outlining austenite grains. Quench cracks were observed to preferentially follow the grain boundaries producing intergranular fracture. (Figures 12 and 13).

RECOMMENDATIONS

1. Examine a sample from a similar leaf spring that gave good service with long fatigue life and which represented AISI 6150 or 6152 steel supplied by other manufacturers.

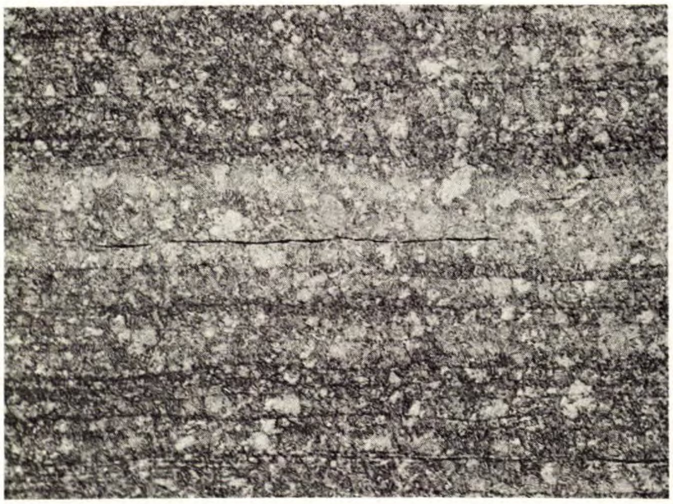
2. For future steel mill production: consideration should be given to
   - use of the minimum specified vanadium content.
   - restriction of silicon to 0.15%-0.25%.
   - elimination of coarse ingot structure and segregation— improvement with respect to inclusion content.
   - the possibility of precipitation of brittle grain boundary constituent whenever this steel is slow cooled from hot hot work temperatures.
   - use of fine grain practice and residual contents of 0.02%-0.03% acid soluble aluminum.

3. Referring to leaf spring manufacture: -
   - Unless precautions are taken to avoid slow cooling and precipitation from forging (rolling) temperatures, reaustenitizing will be necessary to effect solution of this constituent prior to hardening by liquid quenching. (Forging and direct quenching may be possible with competitive steel aluminum-killed and containing minimum vanadium; however, use of the strip from the subject melt will require an austenitizing heat treatment after forging, for dissolution of precipitate and refinement of grain size).
   - Water quenching from the 800°F tempering furnace would appear to be a worthwhile precaution with this heat of steel.

4. Use aluminum-killed steel and form springs at a temperature lower than the grain coarsening temperature.

X100 - Saturated aqueous picric acid and tridecylbenzene sulfonate.

Figure 3. Sample No. 1. - Illustrates long stringer inclusion and banded microstructure.

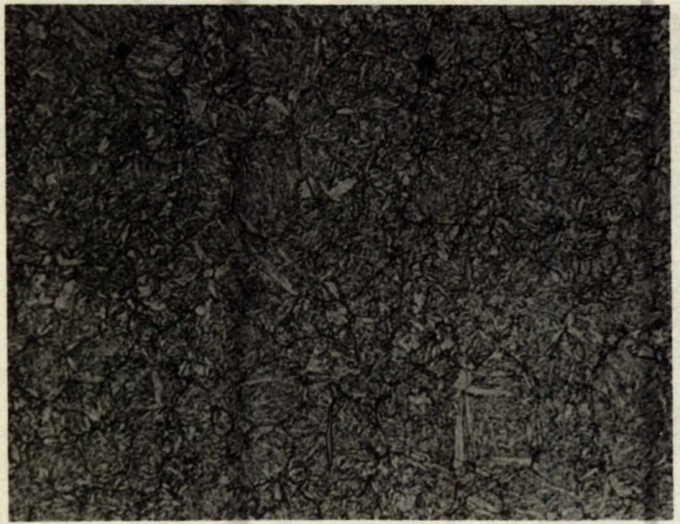


X100 - Same etch as Figure 3.

Figure 4. Sample No. 2 - Stringer inclusions but not as large as those observed in samples No. 1 and 3. Microstructure is banded.

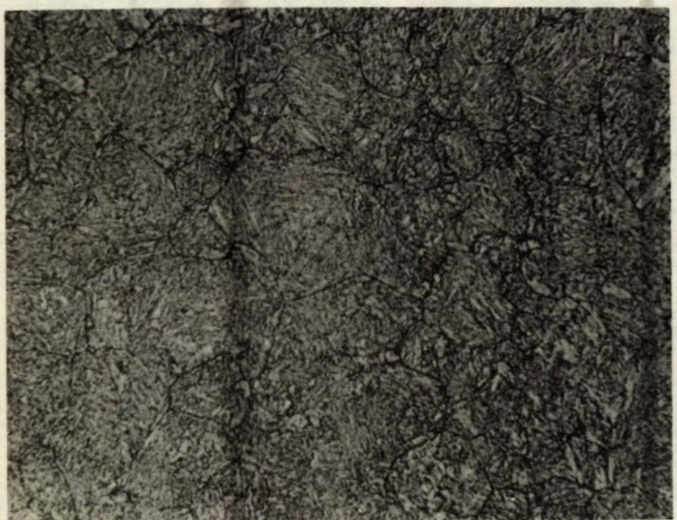
X100 - Same etch as Figure 3.

Figure 5. Sample No. 3 - Large stringer inclusion and pronounced banding is illustrated.



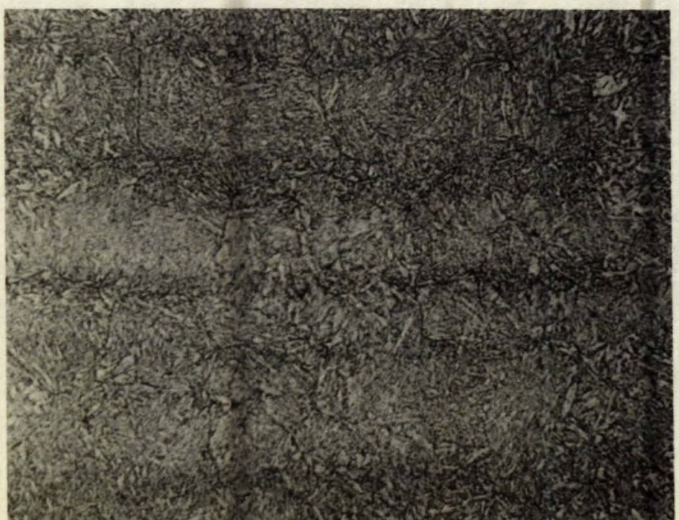
X500 - Saturated aqueous picric acid + tridecylbenzene sulfonate.

Figure 6. Sample No. 1. The austenite grain boundaries are clearly visible, suggesting precipitation of a vanadium phase subsequent to austenitizing. The Shepherd fracture grain size is No. 5



X500 - Same etch as Figure 3.

Figure 7. Sample No. 2 - Austenite grain boundaries are outlined.



X500 - Same etch as Figure 3.

Figure 8. Sample No. 3 - Austenite grain boundaries are outlined, The grain boundaries are not as plainly visible in this sample as in samples No. 1 and 2.

Figure 9. Sample No. 1. Reheat treated; normalized 1650°F (1 hr); oil quenched from 1550°F. Tempered 700°F; water quenched - R_c 50 Shepherd grain size is No. 8. The banding is eliminated or masked. No trace of grain boundary constituent was observed.

X500 - Same etch as Figure 3.

Figure 10. Sample No. 2. Reheat treated as in Figure 9. (R_c 50). The banded structure and grain boundary constituent appear to have been eliminated.

X500 - Same etch as Figure 3.

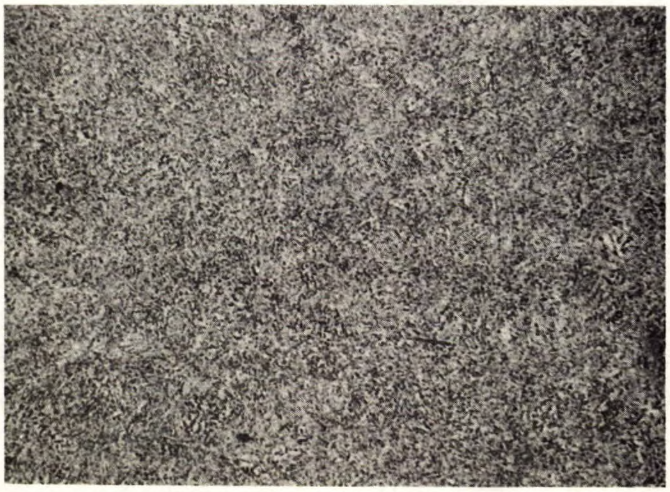
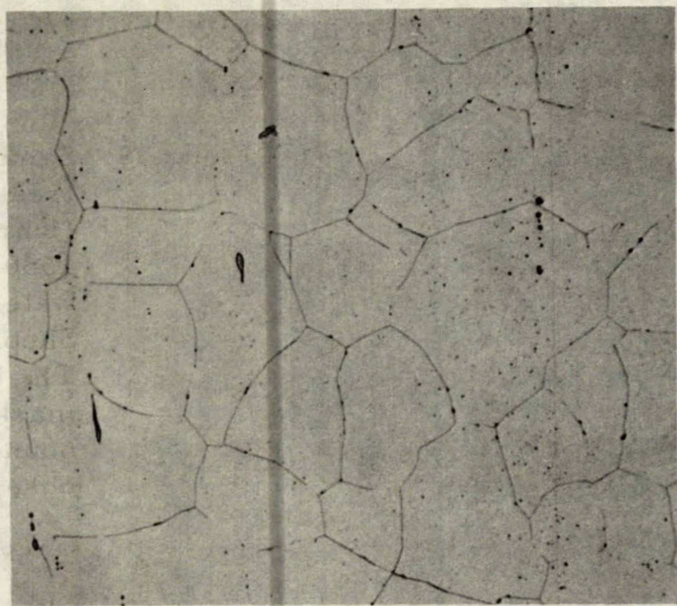


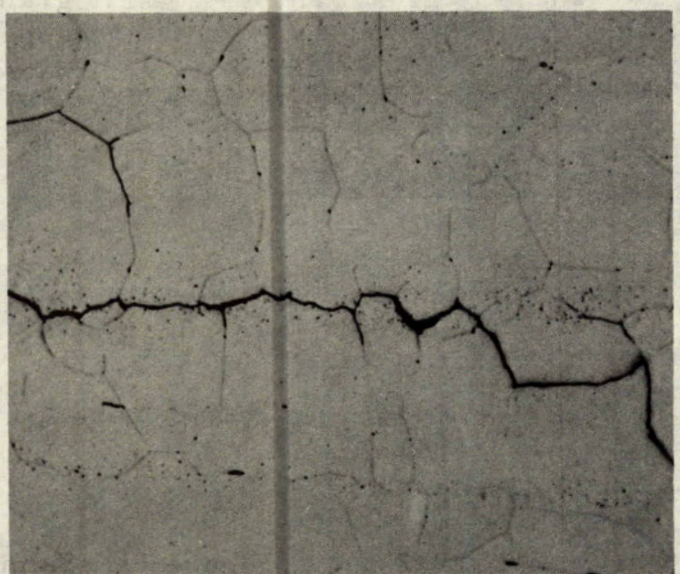
Figure 11. Sample No. 3. Reheat treated as in Figure 9 (R_c 50). Charpy V-notch fractures were much finer grained than those observed in the as-received springs.

X500 - Same etch as Figure 3.



X500 - Etched in saturated aqueous picric acid + tridecylbenzene sulfonate.

Figure 12. Metal from spring No. 2. Reheat treated to eliminate as-received precipitate then heated to 2000°F, slow cooled to 1600°F and water quenched. The grains are outlined with a continuous, brittle film believed to be vanadium carbide.



X500 - Etched in saturated aqueous picric acid + tridecylbenzene sulfonate.

Figure 13. Metal from spring No. 2. As in Figure 12 reheated to 2000°F, slow cooled to 1600°F and water quenched. A quench crack was observed (due to the severity of the water quench from 1600°F used to preserve the 1600°F structure for metallographic examination) and followed a completely intercrystalline path demonstrating the embrittling effect of the grain boundary constituent in coarse grained steel.

APPENDIX

Semi-Quantitative Spectrographic Analyses (Per Cent)

Element	Sample No. 1	Sample No. 2	Sample No. 3
Manganese	0.85	0.85	0.87
Silicon	0.37	0.46	0.50
Chromium	0.95	1.07	1.10
Vanadium	0.22	*0.43	*0.39
Aluminum	0.006**	0.006**	0.005**
Copper	0.18	0.19	0.22
Nickel	0.04	0.06	0.07
Molybdenum	0.010**	0.009**	0.010**
Arsenic	ND	ND	ND
Antimony	ND	ND	ND
Tin	0.007	0.01	0.009
Titanium	0.0005	0.0006	0.0006

* Average vanadium content by wet analysis was 0.22% in all samples. The spectrographic results tend to be higher.

** Check analyses made on drillings.

The residual contents of aluminum, nickel, molybdenum, arsenic, antimony, tin and titanium are very low.