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A STUDY OF SEVERAL COMMERCIAL STEELS IN THE RED SHORT RANGE

BY

JORGE ALBERTO ESPANA

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THES IS

submitted to the faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI

in partial fulfillment of the work required for the

Degree of

MASTER OF SCIENCE, METALLURGY MAJOR

Rolla, Missouri

1962

Approved by

advisor)

 \mathcal{O}_{i}

ABSTRACT

The red short phenomenon present in steels, was studied by means of torsion tests at temperatures between 800 and 1200° C. Cold drawn bars of different compositions of steels were tested, and the number of turns to failure and maximum twisting torque were plotted versus temperature.

All the steels tested, exhibited to have a certain degree of red shortness. By comparing the curves and the respective chemical analyses, the detrimental effect of sulphur and lead and the beneficial effect of manganese, was established.

Microscopic examinations revealed the intergranular nature of the cracks in the red short range. Sulphur and oxide distribution were also studied by macroscopic prints. The results of the tests were in accordance with the behavior predicted by the liquid film theory.

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FOREWORD

The problems related to hot shortness have been the subject of a large amount of research and discussions. Numerous investigators studied them from different approaches, and the principles of these phenomena have been determined.

It was the purpose of this investigation to add some data to that already existent along with the enunciation of facts revealed by the study that were not openly disclosed by previous works.

The author acknowledges and appreciates the helpful cooperation received from some professors and graduate students during the performance of this research.

He is indebted to Professor Dr. D. S. Eppelsheimer, who acted as advisor, suggested the subject of this thesis and contributed with his thoughtful guidance throughout this investigation.

Thanks are due to Dr. A. W. Schlechten, Chairman of the Department of Metallurgical Engineering, and Professor R. F. Davidson, Chairman of the Department of Mechanics, for their interest in securing all the necessary services to make this work possible.

The assistance, advice, and criticism from Dr. W. A. Frad and Dr. H. P. Leighly is deeply appreciated.

The author wants to express his gratefulness to Professor R. V. Wolf, who devoted his time to review and correct the manuscript and for his constructive criticism.

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CHAPTER I

THE PROBLEM AND DEFINITIONS OF TERMS USED

For many years the problems related to hot workability of metals, especially steels, have attracted the attention of numerous investigators. In spite of the large amount of research done in the field, several phenomena are not yet completely understood.

A. THE PROBLEM

Statement of the problem. In operations like hot rolling or forming, steels are processed in the plastic state at elevated temperatures. The higher this temperature of working, the more plastic the steel should become, until it reaches a temperature at which a critical condition related to ductility is present, to such an extent that can lead to breakage of the piece under extreme pressures. The appearance of cracks other than those normally expected, such as the ones originated during solidification or reheating of the ingot, are evidence of this state.

In some cases the original ingot size is regarded as a cause of cracks, for cracks were found to occur more frequently the leavier the original ingot.¹ The formation of the

L. N. Sokolov, "Surface Defects on Rolling Mill Rolls and Heavy Forgings," <u>Kuzenechno-Stampovochnoe</u> <u>Proizvodstvo</u>, No. 8, 1959, pp. 4 et seq.

duplex inclusion of (Mn, Fe) S and "glass", and its deformation after rolling and heating has been shown by Baeyertz.² She states that in multiple phase inclusions, the phases may have different plasticities at rolling temperature and deform accordingly.

During the last 40 years, alloying elements have been added to steels in considerable amounts. Most of them have not markedly affected hot worksbility. Sulphur has been consistently blamed as the cause of the mentioned brittleness in steel while being hot worked, and in numerous cases oxygen has been also made responsible for the same effect. However, this brittle behavior is also reported to be present in pure iron and occurs in Armeo iron between 850 and 1100°C.³

In other words, either inclusions, impurities or changes within the microstructure, or combinations of them may be causes of brittleness of the steel while it is deformed at elevated hot working temperatures.

Importance of the study. Although the effect of several factors on the properties of forged steel are well known, the best conditions for hot working operation itself are still determined by rule-of-thumb.

²M. Baeyertz, <u>Nonmetallic Inclusions in Steel</u>, p. 95. ³Gastone Guzzoni, <u>Gli Acciai Comuni e Speciali</u>, p. 154.

The losses and return of materials from rolling mills and the costly and time-consuming works trials on new or modified steels make any effort to predict the behavior of steels under hot working conditions worthwhile.

The still existing discrepancy between reliable opinions on some facts related to the problem, leaves room for further study of the phenomenon. (cf. Chap. II)

The presence of a critical temperature in steel, in regards to its ductility in the hot working range, is a fact. The question arises as to its cause and its relationship to the proper maximum processing temperatures.

B. DEFINITIONS OF TERMS USED

The critical brittleness of steels, present at a certain hot working temperature in the vicinity of the A₃ transformation temperature is generally called hot shortness and more specifically red shortness.

The following definitions are given by the Metals Handbook:

hot shortness. Brittleness in metal in the hot forming range.

hot working. Deforming metal plastically at such a temperature and rate that strain hardening does not occur. The low limit of temperature is the recrystallization temperature.

This investigation is related to a study of hot shortness and it becomes necessary to point out a distinction between this and other terms sometimes used for similar purposes.

⁴ Metals Handbook, 8th ed., Vol. 1, 1961 "Hot tearing" refers to a failure on the part of the metal to behave in a ductile fashion while being in the temperature range between the solidus and liquidus.

hot tear. A fracture formed in a metal during solidification because of hindered contraction.

Even though in some cases it has been used to indicate hot shortness in general, this term will be left for the problem faced by foundrymen rather than that presented to the forger.

Hot shortness and red shortness could be applied to steels interchangeably; however, while the former has been used for metals in general, the latter has been reserved for steel only.

red shortness. Brittleness in steel when it is red hot.

In several cases, cracks produced during solidification and reheating of ingots were blamed for hot shortness; in some others, "burning" of the steel was considered responsible for failures which were actually caused by hot shortness. The difference should be noted:

Burning of steel has often been considered the cause for this cracking hot shortness even though oxides are not present in the grain boundaries. While burning of the steel is possible and the path of fracture in such cases is intergranular, distinction should be made between steel that has been burned and that which has been processed at too high a temper-

⁵ibid.

⁶Metals Handbook, 7th ed., c 1948.

ature. In the first case, the damage occurs during the heating cycle and the steel is permanently damaged, while the deffects due to processing at too high a temperature occur during the processing operation, although of course the heating has established the conditions for making those deffects possible.

The twist test specimens sometimes used for evaluating hot shortness are usually, as in this investigation, reduced in section at the center of their length, with different investigators varying the length and diameter of this portion. This part of the specimen will be the one in which twisting and failure actually occurs and will be called the "useful" section. Consequently, related terms will also take this name: "useful" length, "useful" diameter.

^{&#}x27;C. L. Clark and J. Russ, "A Laboratory Evaluation of the Hot-working Characteristics of Metals," <u>Transactions</u>, A.I.M.E., Vol. 167, p. 746.

CHAPTER II

REVIEW OF LITERATURE

Much has been written in regard to the problem here studied, with different approaches and degrees of exhaustiveness. Several investigations have been carried out at this school during the last three years, in which the existing literature has been discussed.^{1,2,3}

To avoid overlapping, only the most important articles reviewed before will be mentioned here along with a selection of works gathered by the present investigator.

A. PREVIOUS WORKS

For more than a decade, very important works were performed at Harvard University. Working under the direction of Dr. A. Sauveur during 1922 and 1923, Dr. D. C. Lee studied the discontinuities presented in the tenacity, hardness, and ductility curves by several steels under heating. The most significant findings in this work were related to the blue heat range.⁴ The same investigators,⁵ discovered that when

¹S. Sehgal, Thesis (T1204), M.S.M., 1959, pp. 3-10

²L. Neumeier, Thesis (T1258), M.S.M., 1960, pp. 13-27

³H. Chao, Thesis (T1311), M.S.M., 1961, pp. 10-52

⁴Albert Sauveur, "Steel at Elevated Temperatures," <u>Transactions</u>, A.S.S.T., Vol. 27, pp. 410-48

⁵Sauveur, <u>The Metallography and Heat Treatment of Iron</u> and <u>Steel</u>, pp. 124 et seq. iron or steel undergo their A_3 transformation, they become critically plastic. By heating a bar of iron or of low carbon steel at the center of its length at a temperature exceeding the A_3 point, and then twisting, they found that the largest amount of twisting occurs at points equidistant from the center and at the temperature of A_3 transformation, and not at the hottest point. When heated to the A_3 temperature, the bar twisted at the center. By 1935, improvements were introduced in the machinery and a number of steels were tested under torsion at elevated temperatures.⁶ In this case, a variety of speeds were used within the low speed range (180° per hr. to 3060° per hr.).

When studying steels under tensile tests, a sudden and large decrease in the reduction of area, was found to occur at the vicinity of 1000° C.⁷ Gueussier and Castro⁸ found the same deviations and irregularities presented by common steels in the hot short range, in austenitic steels.

Intergranular brittleness has also been subject of study,⁹ but the attributed causes are not always concomitant

⁶Daniel S. Eppelsheimer, Thesis, Harvard University, 1935. ⁷Alfredo Galassini, Leghe Metalliche e Siderurgia, p. 53. ⁸Andre Gueussier an Rene Castro, "Relations entre les fragilites a chaud et a froi dans les aciers austenitiques du ype 13-8," <u>Revue de Metallurgie</u>, Vol. 55, pp. 107-22.

⁹C. de Beaulieu, "Etude d'un cas de fragilite intergranulaire du fer," <u>Revue de Metallurgie</u>, Vol. 55, pp. 495-9.

with the accepted theories of hot shortness. Analyses of intergranular fractures were presented on a study of steels at elevated temperatures by White et al.,¹⁰ and also by Thielmann and Parker.¹¹

By quantitative hot workability tests, the effects of various elements in this property of steels were studied, however, a well determined law cannot be stated.

The transitory period during hot working has been studied regarding the microstructure of steels. A modification of the structure was found in such period depending on the temper ature and rate of deformation.¹³ A special study on recrystallization under the influence of hot working is also recorded by the same authors.¹⁴

The drop-blow test has also been used as means of determining hot workability of steels. The suitability of the

11 R. H. Thielemann and E. R. Parker, "Fracture of Steels at Elevated Temperatures After Prolonged Loading," <u>Trans-</u> <u>actions</u>, A.I.M.E., Vol. 135, pp. 559-82.

¹²Harry K. Ihrig, "The Effect of Various Elements on the Hot-Workability of Steel," <u>Transactions</u>, A.I.M.E., Vol. 167, pp. 749-77.

¹³C. Rossard and P. Blain, "Evolution de la structure de l'acier sous l'effect de la deformation plastique a chaud," <u>Revue de Metallurgie</u>, Vol. 56, pp. 285-300.

¹⁰A. E. White, C. L. Clark and R. L. Wilson, "The Rupture Strength of Steels at Elevated Temperatures," <u>Transactions</u>, A.S.M., Vol. 26, pp. 52-80.

¹⁴Rossard and Blain, "Influence de la deformation sur la recristallisation de l'acier ferritique a 25% Cr apres deformation plastique a chaud," <u>Revue de Metallurgie</u>, Vol. 57, pp. 173-8.

drop-blow test as compared to the torsion test will be discussed later. It is generally agreed that the former is not as complete as the latter, however, a large amount of data have been obtained by using this procedure.¹⁵

A special rolling mill has been built in Sweden with which the variations of specific pressure of rolling with temperature for different deformations and thicknesses, was determined for several steels.¹⁶

A study of the flow of material in different planes perpendicular to the rolling direction, spreading of differently alloyed steels in a 300 mm small section train and a 950 mm blooming train and the influence of deformation temperatures between 900°C. and 1100°C. has recently been done in Germany.¹⁷

Significant contributions to the field are due to Rossard and Blain. By means of hot torsion tests with different twisting speeds, they studied the deformation of steel under usual forging temperatures.¹⁸ The influence of different rates of deformation was shown. In these tests, the specimens were twisted to several limits in the plastic range, without reach-

150. W. Ellis, "Further Experiments on the Forgeability of Steel," Transactions, A.S.S.T., Vol. 21, pp. 673-707.

16 Gunnar Wallquist, "Berakning av valstryck och energiforbrukning vid varmvalsning," Jernkontorets Annaler, Vol. 138, pp. 539-72.

¹⁷Walter Grosse and Heinrich Gottwald, "Der Einfluss von Kohlenstoff, Mangan, Chrom, Nickel und Molybdan auf das freie Breiten von Stahlen," <u>Stahl und Eisen</u>, Vol. 79, pp. 866-73.

¹³Rossard and Blain, "Premiers resultats de recherches sur la deformation des aciers a chaud. Mise au point d'un apareillage specialement etudie," <u>Revue de Metallurgie</u>, Vol. 55, pp. 573-94.

ing the failure point. The graphic determination of the transitory phenomena during hot working of steels, was also studied.¹⁹

The effect of strain rate in metals was the subject of a recent research carried out by Kattus. He reported that this effect becomes greater at elevated temperatures.²⁰

Much work has been devoted to the determination of the effect of different components in the hot workability of Fe-C alloys. There is general agreement on the detrimental effect of sulphur and the counter effect of manganese. By using compositions low in manganese with different sulphur contents, Anderson et al. studied the influence of sulphur by means of twist tests.²¹

B. THE LIQUID FILM THEORY

Equi-cohesive temperature. The liquid film theory provides means of explanation of the hot short phenomenon. It is based on the concept of equi-cohesive temperature introduced by Jeffries as early as 1917. The original work could not be more clear or accurate when defining this concept:

¹⁹Rossard and Blain, "Procede de determination graphique des phenomenes transitoires cours de la deformation des aciers a chaud," <u>Revue de Metallurgie</u>, Vol. 55, pp. 595-8.

²⁰j. R. Kattus, "The Effects of Strain Rate and Temperature on Metals," <u>Metal Progress</u>, Vol. 30, No. 6 Dec. 1961, pp. 85-9.

²¹C. T. Anderson, R. W. Kimball and F. R. Cattoir, "Effect of Various Elements on Hot-Working Characteristics and Physical Properties of Fe-C Alloys," <u>Transactions</u>, A.I.M.E., Vol. 197, pp. 525-9.

According to the amorphous cement theory, in most metals the amorphous phase is harder and stronger, that is, more cohesive, than the crystalline phase at room temperature or other relatively low temperatures, whereas at high temperature the reverse is true. Obviously, if this is true there must be some intermediate temperature in any given metal at which the cohesion of the amorphous and crystalline phases is the same. I will refer to this temperature as the "Equi-Cohesive Temperature."22

influence of sulphur. The role played by some components in hot shortness of steel should be reviewed.

Sulphur diffuses into iron preferentially along the grain boundaries. When it enters the iron lattice, it diffuses laterally from the grain boundaries but keeps a high concentration in the vicinity of the boundary.^{23,24}

By autoradiographic techniques and using radioactive sulphur, the migration of this element in steel has been studied,²⁵ while the classic macrographic and micrographic examinations fail to reveal the local variations of the sulphur content or satisfactorily resolve the inclusions.

²⁵A. Kohn, "Etude de la migration du soufre dans l'acier," <u>Revue de Metallurgie</u>, Vol. 55, pp. 265-74.

²²Zay Jeffries, "The Amorphous Metal Hypothesis and Equi-Cohesive Temperatures," Journal of the American Institute of Metals, Vol. 11, p. 311.

²³N. G. Ainslie, R. E. Hofman and A. U. Seybolt, "Sulfur Segregation at Alpha-iron Grain Boundaries I," <u>Acta Metal-</u> <u>lurgios</u>, Vol. 8, pp. 523-7.

²⁴N. G. Ainelie, V. A. Phillips and D. Turnbull, "Sulfur Segregation at Alpha-iron Grain Boundaries II," <u>Acta</u> Metallurgica, Vol. 8, pp. 528-38.

It was confirmed that up to 0.007 per cent sulphur in pure iron is totally soluble in iron, but when carbon is introduced, the sulphur appears almost entirely at the grain boundaries.

When manganese is present, it separates the sulphur in the form of a distinct phase. The precipitates rich in sulphur deposit at a lower temperature than that of solidification at the grain boundaries or form inclusions. These inclusions in some cases, are disseminated in the metal preferently in the interdendritic spaces.

According to Sauveur:

The sulphide Fe S exhibits a marked tendency to form continuous envelopes or membranes surrounding each grain of pearlite...probably consisting of a cutectic alloy of iron and iron sulphide (the composition of the cutectic is apparently Fe S 85 per cent, Fe 15 per cent). These membranes being weak and brittle₂₆ impart weakness and brittleness to the steel.

This is the case of forming of steel under elevated temperatures. The iron iron sulphide eutectic is reported to have a melting point of 950°C. to 1200°C. decreasing the strength of the grain boundaries when those temperatures are reached. This is responsible for the appearance of intergranular cracks if deformation exceeds that which the eutectic is capable of withstanding, and may cause eventual breakage of the piece. This could be overcome by the addition of Mn, to

²⁶Sauveur, <u>The Metallography and Heat Treatment of</u> <u>Iron and Steel</u>, p. 90

form MnS with a melting point of 1620° C. or more likely $\text{Pe}_{3}\text{Mn}_{2}\text{S}_{5}$ which has been reported to have a melting point of 1635° C. In this case the equi-cohesive temperature is raised and the hot-workability of the alloy is improved.

The weakening effect and the intergranular cracks have been blamed by some investigators on the less perfectly co ordinated structure of the grain boundary, but the low melt ing point of its components was taken into account.²⁷

The liquid film theory has been also asserted as the cause of hot tearing. 28

Determinations of equi cohesive temperatures. Much work has been done towards determination of equi-cohesive temperatures but neither the tests nor the rates of deformation have been standardized. It must be pointed out that the values of equi-cohesive temperatures are strongly dependent on the rates of deformation imparted to the piece studied. An example of this variation is given by Clark and Russ:

For example, in 0.15 per cent plain carbon steel the location of this temperature under the rates of deformation encountered in creep tests (of the order of 1 percent per 10,000 or 100,000 hr.) is less than 800°F., while in the rupture tests and for fracture times of 1,000 hr. or greater, it is about 900 deg. F. On the other hand, under the rates of deformation usually employed in tensile tests, this temperature is of the order of 1500 deg. F.²⁹

²⁹Clark and Russ, op. cit., p. 757.

²⁷K. T. Aust and B. Chalmers, "Energies and Structure of Grain Boundaries," <u>Seminar on Metal Interfaces</u>, p. 158.

²⁸W. S. Fellini, "Strain Theory of Hot Tearing," Foundry, No. 80, pp. 125-7.

A special tensile test with controlled speed gave values of 650°C. to 750°C. for the equi-cohesive temperature of steel, but it was made clear by the authors that it would change depending on the rate of deformation.³⁰

Jeffries³¹ determined the equi cohesive temperatures for different metals obtaining the results shown in Table I. Hair pin tests were used for such determinations in which the rate of deformation is slow.

The phenomenon of change in elastic limit and hence ductility of a metal severely worked at different temperatures is graphically shown in Fig. 1.³²

Finally, one more fact should be kept in mind while testing a metal under tension. The cross section of the test piece is smaller than the area presented by the grain boundaries. Therefore, intercrystalline fracture will be present when the temperature is such that the cohesion of an area of the amorphous phase equal to the area of fracture along grain boundaries is less than the cohesion of an area of the crystalline material equal to the cross section of the test piece.

³⁰R. Tamhankar, J. Plateau and C. Crussard, "Etude de la deformation plastique a chaud d'un fer doux et d'une austenite estable au nickel-chrome," <u>Revue de Metallurgie</u>, Vol. 55, pp. 383-400.

³¹ Jeffries, op. cit.

³²Jeffries, "Effect of Temperature, Deformation and Grain Size on the Mechanical Properties of Metals," <u>Transactions</u>, A.I.M.E., Vol. 50, p. 530.

TABLE I

EQUI-COHESIVE TEMPERATURES DETERMINED BY JEFFRIES BY MEANS OF HAIR PIN TESTS

Metal	Diameter	Equi-cohesive temperature
an an an an Alban an	ln.	deg. C.
Iron	•007	550 - 600
Tungsten	.007	1350
Silver	.0075	250 - 275
Gold	.0085	275 - 300
Platinum	.0075	525 - 550



Fig. 1 - Equi-cohesive temperature as shown by Jeffries.

CHAPTER III

THE TEST

Torsion tests at elevated temperatures were performed in this investigation to evaluate and compare hot working characteristics of several steels. This has been considered more suitable than the other types of test sometimes used for the same purpose. To show adequate reasons for this selection, a brief analysis of the most used methods other than the twist test is given here.

There have been several tests at elevated temperatures tried in previous investigations for laboratory evaluation of hot workability of steels. Bend tests were used, in which the samples were subjected to bending back and forth over the sharp end of an anvil or in other cases, bent in one direction to a certain determined degree.

A number of tensile tests have also been used. Some of them under impact loading, some others with a controlled rate of deformation. Tension, temperature and reduction of area were variables measured in these tests.

In a report of work on hot-hardness of high speed steels, Harder and Grove¹ propose the mutual indentation method of determining the hardness of metals as developed by Cowdrey²

¹Oscar E. Harder and H. A. Grove, "Hot-Hardness of High-Speed Steels and Related Alloys," <u>Transactions</u>, A.I.M.E., Vol. 105, p. 123.

²I. H. Cowdrey, "Hardness by Mutual Indentation," <u>Proceedings</u>, A.S.T.M., Vol. 30, pp. 559-70.

as a means of determining the suitability of materials for service at elevated temperatures and in selecting hot-working temperatures for metals and alloys. In this test, two cylinders of the material to be tested are blaced parallel and subjected to a predetermined load for 30 sec. By measuring the area of the flattened or indented surface, the cylinder hardness was determined. Tests with spherical indentation were used in other investigations. Hardness can be measured at elevated temperatures with the same accuracy as it can at room temperature.³

Drop forging tests were widely used. Obviously, this test provides conditions more similar to those of hot working than the methods mentioned above. Two different procedures characterize the types of blow test used. In one case, specimens were reduced by several blows to a predetermined percentage of its original height. In the other type, a single blow was imparted to the sample with the amount of energy of the blow remaining constant for each sample studied.

These methods are not accurate determinations of hot working characteristics. In the bend and drop tests, the experiment is performed outside of the heating furnace. Inevitable heat losses preclude an accurate recording of the actual working temperatures. Hardness and tensile tests do not impart the type of stresses to which the steel would be

³Frederik P. Bens, "Hardness Testing of Metals and Alloys at Elevated Temperatures," <u>Transactions</u>, A.S.M., Vol. 33, pp. 505-16.

subjected during hot rolling or forming. Measurements of energy are also difficult in some of these tests.

The hot torsion tests is generally accepted as the most appropriate for evaluation of hot working characteristics. One reason for using this test is the simplicity of the apparatus. By means of a d.c. motor or a gear transmission the rate of deformation can be varied and controlled. Since the specimen is subjected to torsion, the predominating stresses are in shear.

The stress distribution over the section of a test piece subjected to torsion and tension is of the form shown in Fig. 2,⁴ the tensile stress being a maximum at the center where the shearing stress is zero. Since the value of the tensile stress is at a minimum at the outside of the specimen, there may be a transverse flow effect, but it is not likely to cause premature failure.

Numerous machines have been used for torsion tests at elevated temperatures, all of them based on the same principle with differences only in the recording mechanisms or speed reduction systems. A very modern device was built at the IRSID (Institut de Recherches de la Siderurgie), Fig. 3. It is capable of developing speeds of 0.1 to 1,000 r.p.m., it has an electromagnetic clutch that can stop the test almost instantaneously and could run time controlled tests to a minimum of 0.1 sec. Torques are measured in a range of 0.07 to 70 kg.cm.

⁴A. Nada1, <u>Plasticity</u>, p. 216.



Fig. 2 - Plastic stress distribution in solid cylinder under combined torsion and tension.



Fig. 3 - Torsion test machine built at the IRSID.

Rossard and Blain,⁵ who designed this torsion test machine, used a very ingeneous method for registrating measurements also found in other investigations performed in France.⁶ By placing together a chronograph, a turn counter and a torque indicator in front of a movie camera, almost every instant of the test is recorded in the film.

A. SPECIMENS

Design. The size and shape of the specimens for hot torsion tests have not yet been standardized, and they usually vary from one investigator to another. Anderson et al.,⁷ Ihrig,⁸ and Clark and Russ⁹ used cylindrical bars without any reduced section, the size varied from 3/8 in. to 5/8 in. in diameter. Several investigators used specimens with a reduced section. Sauveur selected bars of 1/2 in. in diameter with a useful section of 1/4 in. in diameter.¹⁰ Hughes ¹¹ had $1\frac{1}{2}$ in. useful length with a diameter of 3/8 in. in a 9/16 in. diameter bar. Chao used a 5/8 to 3/4 in. diameter bar with a total

⁵Rossard and Blain, "Premiers resultats de recherches..." ⁶M. Very, "Etude de la forgeabilite de l'acier," <u>Circul-</u> <u>aire d'informations techniques</u>, Centre de documentation siderurgique, Vol. 12, pp. 783-800.

⁷Anderson, Kimball and Cattoir, op. cit.

³Ihrig, op. cit.

⁹Clark and Russ, op. cit.

¹⁰Sauveur, "Steel at Elevated Temperatures."

11D. E. R. Hughes, "The Hot-Torsion Test for Assessing Hot-Working Properties of Steels," <u>Journal of the Iron and</u> <u>Steel Institute</u>, Vol. 170, pp. 214-20. length of 17 to 19 in. and a 40 mm long useful section of 10 mm diameter.¹² Rossard and Blain used a 12 mm diameter bar with a length of 356 mm and a 50 mm useful length and 6 mm useful diameter. A design similar to the last one was adopted for this investigation, and it is shown in Figs. 4 to 6.

The use of cylindrical bar without a reduced section is seriously objectionable. The heat transfer along the bar originates differences in temperature from one point to another, even in the portion that is inside the furnace. Thus, preferential zones of twisting will exist and the actual length that undergoes deformation could not be precisely determined. By the use of a reduced section, this effect is minimized. The useful section is placed in the center of the furnace and a considerable length of the large diameter portion of the bar is also inside of the heating device.

The selected design was found to have advantages over the others mentioned of the same type, because having a larger useful length with a smaller diameter the number of twists to failure will be larger and greater accuracy of measurement can be accomplished.

<u>Composition</u>. Five different commercial steels were selected for the tests, with compositions from which a different behavior in the red-shortness range would be expected. These steels are described in Table II.

¹²Chao, op. cit.





Fig. 5 - Specimens for the twist tests.



Fig. 6 - Close up of the useful section.

TABLE II

COMPOSITIONS OF SPECIMENS

Steel	AISI	C	iin	P	S	Other	
W	C 1113 MX	0.10 - 0.16	1.00 - 1.30	0.040 max.	0.24 - 0.33	fan Breikinne in Gronn gebrûk fin oarden in Staart yn	
0	C 1213	0.13 max.	0.70 - 1.00	0.07 - 0.12	0.24 - 0.33		
В	°C 12 L 14	0.15 max.	0.80 1.20	0.04 - 0.09	0.25 - 0.35	0.15 - 0.35 Fb	
G	C 1117	0.14 - 0.20	1.00 - 1.30	0.040 max.	0.08 - 0.13		
¥	C 1018	0.15 - 0.20	0.60 - 0.90	0.040 max.	0.050 mex.		

* Commercial name: Ledloy 300

Most of the specimens belonged to the free machining type, some were rephosphorized and resulphurized. Lead has been added to one of them in an amount of 0.15 to 0.35 per cent in order to improve machinability. The Mn/S relation varied from 3 to 15 and P has been kept under 0.04 per cent in some compositions, but is close to 0.1 per cent in those of the rephosphorized type.

Micrographs "as received" are shown in Figs. 7 to 10. Samples were all cold drawn bars of $\frac{1}{2}$ in. diameter from which the specimens were cut and machined to the selected design.

B. APPARATUS

A bench type Tinius Olsen torsion testing machine was used. Samples to be twisted are held between a loading and a weighing jaw, the distance between jaws being adjustable. A load indicator placed on the weighing head shows the torque resistance of the sample directly in inch pounds. This torque indicator, designed to measure maximum torque during testing, was modified for the purpose of this investigation. By making some adjustments in the mechanism of the indicator and adding a second pointer to the scale, instantaneous and maximum torques were both measured. This provided means of readily determining the failure of the sample.

A motor provides a single speed of 180 deg. per min. and rotation can be read on the loading head in degrees or by means of a turn counter in 0.01 turn. The machine with the addition of the heating furnace is shown in Fig. 11.



Fig. 7 - Steel W, transverse section; 400 x; 35 Nital.



Fig. 8 - Steel 0, transverse section; 400 x; 3% Nital.


Fig. 9 - Steel G, transverse section; 400 x; 3% Nital.



Fig. 10 - Steel B, transverse section; 400 x; 3% Nital.



Fig. 11 - The torsion test machine during operation.

A Kanthal tube type furnace was used as a heating unit. It was placed on a table especially built to allow it to slide back and forth, and transversally during placing and removal of the specimens, and to allow alignment with the axis of rotation of the torsion machine. The furnace has a built-in thermocouple with a temperature indicator, a signal lamp, and an energy regulator. This last device provides means of regulating temperature by automatically opening and closing a relay in the furnace circuit at proper time intervals. The heating chamber dimensions are: 2 3/4 in. in diameter and 7 7/8 in. long. A protecting tube was placed and another thermocouple was used to indicate the temperatures in a point close to the useful section of the bars tested. This temperature was recorded by means of a Bristol Dynamaster potentiometer. The whole set-up is shown in Fig. 12.

C. PROCEDURE

The specimen was placed in the furnace from the weighing side of the machine and clamped in the loading head. The furnace openings were closed with asbestos to reduce oxidation of the sample. The weighing head was then brought into position, but the piece was left unclamped so that free expansion during heating would be allowed.

The specimens were soaked for 20 to 30 min. at the intended temperature of the test to attain a uniform temperature and to eliminate the influence of previous work to which they had been subjected. The temperature of the furnace was controlled



Fig. 12 - Set-up for torsion tests.

at a point close to the heating elements by the built-in thermocouple and measured in a zone close to the center of the useful section of the specimen by the potentiomemeter.

When the specimen was ready it was clamped in the weighing head, the load scale was adjusted to zero and the torsion was started for a few degrees by hand operation and then the motor started.

The end of the test was indicated by the torque scale, when the pointer indicating actual torque returned to zero. The machine was stopped and the number of twists and maximum torque recorded. The specimen was removed and cooled in the air.

CHAPTER IV

EXPERIMENTAL RESULTS

A. TORSION TESTS

The tests were conducted at temperatures between 800°C. and 1200°C. The results obtained are summarized in Table III in which temperature, maximum torque and the number of turns to failure are recorded. Some specimens failed at the neck of the useful section. Such values were not taken into account for the evaluation.

The temperatures given are those obtained with a thermocouple near the center of the useful section. Since an appreciable length of the large diameter portion of each end of the specimen was inside the furnace, it is assumed that no temperature gradient existed over the length of the useful section.

Oxidation occured during the tests and its influence was regarded as a possible source of error in the results. Since the shear stress imparted to the piece was at a maximum at the surface and zero at the center, the embrittlement of the outer layers by oxidation, and the consequent decrease of the actual twisting diameter, could cause error in the test values. For that reason, tests were run in which different amounts of oxidation were imparted. By leaving openings in the furnace and increasing the soaking times, a greater oxidation was attained. To minimize the oxidizing

TABLE III

Specimen	Twisting Temperature	Maximum Torque	No. of twists
	deg. C.	inch-lb.	to failure
B-80	808	12	10
B-90	915	13	8.5
B-100	995	12	6.5
B-110	1125	7.5	8
B-120	1175	6.5	9
G-80	815	21	14.5
G-90	910	12.5	18
G-100	995	10	14.5
G-110	1075	7	18
G-120	1175	5	19.5
0-80	815	14	18
0-90	915	12.5	11
0-100	990	12	11
0-110	1085	7	20.5
0-120	1160	5	15
W -80	810	15	11
W -90	908	17	10.5
W -100	980	12	11
W -110	1080	8	17
W -120	1170	7.5	23
Y-80	810	18	23. <u>5</u>
Y-90	915	11	22
Y-100	1015	7	21
Y-110	1080	6	24
Y-120	1125	5.5	25

TWIST TEST RESULTS

effect, tests were run in the presence of a reducing agent (graphite) following a technique that is used in some heat treatments of steel. The difference obtained in torque values and number of twists to failure were very small and inconsistent. From this it was concluded that the oxidation to which the samples were subjected by following the procedure explained in Chap. III, was not leading to incorrect results. Specimens showing differences in oxidation are exhibited in Fig. 13. Typical samples of tests run at temperatures between 900°C. and 1200°C. are shown in Figs. 14 to 17.

From the data obtained, curves were plotted for each steel examined. The number of twists to failure and the maximum torque versus the twisting temperatures are shown in the graphs, Figs. 18 to 22.

All the steels studied presented red shortness to a certain extent. The most definite range of brittleness was showed by steels 0 and B. The former showed a large decrease in the number of turns to failure in the range between $900^{\circ}C$. and $1000^{\circ}C$. Steel B did not present such a distinct decrease in the number of turns to failure as steel 0, but the phenomenon was present for a much wider range of temperature. Even the number of twists at $1200^{\circ}C$. was less than the necessary to produce failure at $800^{\circ}C$.

According to Clark and Russ, 1 for the rate of deformation involved in the particular twist test, the temperature of

¹Clark and Russ, op. cit.



Fig. 13 - Different degrees of oxidation shown by specimens tested at 800°C.



Fig. 14 - Steels G, Y and W tested near $900^{\circ}C$.



Fig. 15 - Steels Y, B and W tested near 1000°C.



Fig. 16 - Steels G, B and O tested near 1100°C.



Fig. 17 - Steels B, W and O tested near 1200°C.











maximum twists is the equi-cohesive temperature of the steel under test. In this case, most of the steels studied decreased the twists to failure above the first temperature ($800^{\circ}C.$), and only one case shows a maximum around $900^{\circ}C.$ This is possible due to low speeds used in the test. (cf. Chap. II)

The influence of the rate of deformation in the values of equi-cohesive temperature and results of elevated temperature tests is great and it must always be considered when comparing the results of different investigations. By comparing a large number of tensile tests under high temperatures, Kattus found that more than 30 per cent increase in strength occured in 65 per cent of the specimens and that 45 per cent presented an increase in strength exceeding 100 per cent when strain rate was increased from 0.00005 to 1.0 in. per in. per sec.²

In most of the tests, the torque values decreased with an increase in temperature, following similar curves.

<u>Microscopic examinations</u>. All the types of steel studied were examined under the microscope. Micrographs were taken from some of the specimens.

Inclusions were shown in the grains and along the grain boundaries. The tendency of Fe S to form envelopes surrounding the grains of pearlite was shown under high magnifications and was also observed in several ferritic grain boundaries.

²J. R. Kattus, "Effect of Holding Time and Strain Rate on the Tensile Properties of Structural Metals," <u>Sumposium on</u> <u>Short-time Elevated Temperature Testing of Metals</u>, pp. 105 et seq.

Micrographs of steel B worked under 1000°C. are shown in Fig. 23 and 24. Both were taken along the axis of the bar in the portion of large diameter, close to the useful section. The first one showed that recrystallization of ferrite was completely attained; however, the pearlite grains still showed a preferred orientation along the axis, due to the severe banding in the cold work received before.

A transverse section at 6 mm from the plane of failure was taken for a steel W worked at 300°C., Figs. 25 and 26. Some examples of MnS inclusions, with a clear dove or gray color, were shown while several of them were yellowish tinted indicating that there, the MnS is not free from a certain amount of FeS. The influence of the severe shear stresses is also noticed in the microstructure.

In Figs. 27 and 28 longitudinal section of a specimen are shown under 100 x and 400 x magnifications. These were taken in the useful section of the bar and no trace of the previous cold work was left.

A typical intergranular crack is shown in Fig. 29. Most of the cracks found in the specimens started in the outside, where the deformation is more severe, but a few internal cracks also occured. The cracks were found to be in planes perpendicular to the axis, which was expected from the type of work to which the steels were subjected. The intergranular nature of the crack is readily shown by the mentioned micrograph. In the case of the internal crack also shown in it, the presence of an inclusion along part of its edges is notice-



Fig. 23 - Specimen B-100, longitudinal section; 100 x; 3% Nital.



Fig. 24 - Specimen B-100, longitudinal section; 400 x; 3% Nital.



Fig. 25 - Specimen W-80, transverse section at 6 mm from the plane of failure; 100 x; 3% Nital.



Fig. 26 - Specimen W-80, transverse section at 6 mm from the plane of failure; 400 x; 5% Nital.



Fig. 27 - Specimen Y-30; longitudinal section; 400 x; 3% Nital.



Fig. 28 - Specimen Y-80; longitudinal section; 400 x; 3% Nital.



Fig. 29 - Typical intergranular crack, specimen B-100, longitudinal section; 400 x; 3% Nital.

able.

<u>Macroscopic examinations</u>. The steels were also examined after testing, by macrographic techniques.

Sulphur prints were made on longitudinal and transverse sections of the specimens as shown in Figs. 30 and 31.

The distribution of sulphides was even and no areas of concentration of such constituent was revealed by the prints. The appearance of certain white spots in the prints does not indicate the absence of sulphide in those zones. The inconsistency of their location in a large group of prints made for the same specimen indicated so. They were originated by failure of good contact between the surface of the metal and the paper due to low pressures applied. When printing on glossy type paper, greater pressures lead to a dark tinted print with lack of resolution. Thus, evenness of the print must be sacrified to obtain more revealing details. The procedure followed is explained in the appendix.

Oxide prints were done following a method based on that proposed by Niesner.³ The depth of oxidation of the specimens after testing was shown by these prints. In Fig. 32, a thin layer of oxide covering the surface of the steel is revealed.

³F. Newirth, R. Mitsche and H. Dienbauer, "Anwendbarkeit des Oxydabdruckverfahrens nach M. Niesner," <u>Arch</u> <u>Eisenhutenv.</u>, Vol. 13.



Fig. 30 - Specimen 0-80; sulphur print; longitudinal section.



Fig. 31 - Specimen W-80; sulphur print; transverse section.



Fig. 32 - Specimen 0-110; oxide print; longitudinal section.

CHAPTER V

SUMMARY AND CONCLUSIONS

A. SUMMARY

The red shortness phenomena have been studied by means of a torsion test at elevated temperatures. Several commercial steels were studied including structural steel (1018), rephosphorized and resulphurized, and lead bearing. The presence of lead, which according to the results is important to red shortness of a steel, can be originated by two reasons: (1) Additions with the purpose of improving machinability and (2) when lead treatment is used for copper removal.

Due to the impossibility of removing copper under the reducing conditions of the blast furnace or the oxidizing influence of the open hearth or bessemer processes, lead treatments have been tried.^{1,2} By using this method, copper is removed more or less effectively but a trace of lead remains in the steel. The balance is shown in Table IV.³ Since this procedure is not commonly used, it can be considered that the additions to improve machinability are the main sources of lead in steel.

¹Orkla Grube - Artiebolag, British Patent No. 239,768. ²Y. Ogawa, Japanese Patent No. 1950-16.

³F. C. Langenberg and F. W. Lindsay, "Removal of Copper from Iron-copper-carbon Alloys," <u>Transactions</u>, A.I.M.E., Vol. 200, pp. 967 et seq.

TABLE IV

Ana	lyzed	Iron	Ana	lvzed	Iron		Approximate
Before Lead			After Lead			Temperature	
No	Cu	C	Cu	% C	Pb of	Wt of Pb/Wt (Fe Cu C)	Before Pb Addition, °C
1	0.67	3.86	0.26	4.31	0.052	1.18	1445
3	0.37	3.54	0.08	4.31	0.017 0.010	5.50	1455 1573
8	0.21	2.76	0.07	3.73	0.035	1.66	1390
10	7.17	2.27	1.86	4.21	0.017	1.53	1395
32	8.48	2.19	1.67	4.21	-	1.53	1470

RESULTS OF LEAD TREATMENT

The presence of oxygen has been considered a cause of redshortness. However, for many years the determination of oxygen in steel has been difficult.⁴ It was found that it is detrimental when present in amounts over 0.10 per cent, but no effect was recorded when sulphur content of the same steels was kept under 0.01 per cent.⁵

Curves were drawn with the values obtained from the tests, showing the variations of torque and number of twists to failure with the temperature.

Microscopic examinations revealed intergranular fractures and macroscopic prints disclosed an even distribution of sulphur. A thin oxide layer covering the specimen was shown by oxide printing.

B. CONCLUSIONS

Red shortness was found to be present to a certain degree, in each one of the steels studied. The results of the tests illustrate the typical influence that sulphur and manganese are expected to exert on the steel. The lowest values in the number of twists to failure were present in those steels that were resulphurized and lead bearing, which presented the lowest values of MnS ratio.

J. R. Cain, "A Critical Study of the Ledebur Method for Determining Oxygen in Iron and Steel," <u>Bureau of Stand-</u> ards, Technologic Paper No. 118.

⁵Cain, "Influence of Sulphur, Oxygen, Copper and Manganese on the Red Shortness of Iron," <u>Bureau of Standards</u>, Technologic Paper No. 261.

The high affinity of Mn and S is well known and theoretically would be enough to have a Mn (atomic weight 55) content of about the double of S (atomic weight 32) to form MnS and eliminate completely the FeS. This does not hold true in practice, and even when the Mn/S ratio is found to be higher than 2, the existence of FeS is not eliminated. This sulphide will remain, forming a solid solution with the manganese sulphide. The detrimental effect on the hot short behavior of steels attributed to oxygen is due to the fact that Mn would be oxidized, reducing its preventive effect when sulphur is present in high values.

Lead reduces the hot workability of steel when present in sufficient amount. Its detrimental effect is additional confirmation of the liquid film theory. Lead and iron are insoluble in the liquid and in the solid state. Even though it is not seen by microscopic examination unless very high magnifications are used, it should be located along with other impurities at the grain boundaries. Because of its location, its low melting point affects the values of the equi-cohesive temperatures.

When using the torsion test to determine hot working characteristics, the rate of deformation should be considered a variable of the same level of importance as the temperature.

Finally, the determination of a peak in the curves of twists to failure and the beginning of the red short range give the indication of the maximum temperature to which that steel can be worked. This does not mean that the steel should

invariably be worked at the highest possible temperature at which it will present more plasticity, for the final temperature of working should be taken into account. If the hot working is finished at too high a temperature, a coarse grained final structure will be obtained in the product.

SUGGESTIONS FOR FURTHER STUDY

The importance of the rate of deformation in the results of the tests makes it necessary to try different twisting speeds. However, these speeds, as well as other procedures that could bring variances should be standardized. Standardization of the test is the first step to provide the possibility of comparison and correlation between different investigations. The size of the specimens as well as the furnace atomspheres should also be considered when proposing the standardization.

The additional information obtained from other tests, such as tensile or drop blow tests, would provide data which could be usefully considered when evaluating hot workability.

Further study of the constitution and properties of the grain boundaries would lead to a better understanding of the red short phenomena. As it has been said, their influence is prominent in the appearance of red shortness.

By using isotopic sulphur, its distribution can be studied by autoradiographic methods, but not only this element should be considered when studying the grain boundaries.

APPENDIX

MACROSCOPIC PRINTING TECHNIQUES

Sulphur printing.

The surface to be studied was ground on emery paper to 600 grit, and then washed thoroughly with soap solution, rinsed with methyl alcohol, and dried under warm air blast.

Contact printing paper was soaked in a 2 per cent aqueous solution of sulphuric acid for about 4 min. After soaking, the paper was placed on a flat surface with a paper towel underneath to secure a better contact with the metal. Excess of solution on the paper was removed by passing a squeegee roller.

The specimen was then placed in contact with the emulsion for 1 min. applying a moderate pressure. The sulphide regions of the steel will react with the sulphuric acid produing hydrogen sulphide gas. The gas reacts with the silver bromide in the emulsion, depositing silver sulphide. This gives a brown coloring to the paper in the zones where the iron and manganese sulphides are present.

The equations of the reactions are:

$$FeS + H_2SO_4 = FeSO_4 + H_2S$$
 (1)

$$H_{g}S + 2 AgBr \underline{\longrightarrow} Ag_{g}S + 2 H Br \qquad (2)$$

Manganese sulphide reacts in the same way as iron sulphide in eq. (1).

After removal of the specimen, the paper was washed in running water and then fixed permanently in F-5 photographic fixing solution for about 15 min. and finally washed in water for about 1 hour.

Although the entire process can be carried out in day light, it is better to avoid exposing the paper to light for a prolonged period, which can lead to a slight yellow coloring of the white emulsion.

Oxide printing.

A modification of Niesner's method was used for oxide prints.

The photographic paper was first soaked in a 1:20 aqueous solution of hydrocloric acid. After removed from it, the paper was treated in the same manner as in the sulphide printing and placed in contact with the surface which was ground to a fineness of a 600 grit. emery paper. After 1 min. of contact under moderate pressure, the paper was washed and developed in a solution containing 20g. of potassium ferricyanide for about 5 min. A 10 min. wash in running water followed the development and then the print was fixed in F-5 fixing solution and finally washed for 30 min.

In both cases, sulphide or oxide prints, the specimens were repolished after one or two prints.

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