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A STUDY OF HOT SHORTNESS IN STEELS

BY

KOANG-HWA KUNG , 1934

Α

THESIS

submitted to the faculty of the

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degree of

MASTER OF SCIENCE IN METALLURGICAL ENGINEERING Rolla, Missouri

Approved by 2/usihne (advisor)

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INTRODUCTION

A considerable amount of work has been done on the problem of hot shortness in steel. However, the results of these studies have often been contradictory and inconclusive. More work is needed to determine the exact cause of hot shortness and on methods by which it can be avoided. Fortunately, the addition of sufficient manganese to the steel will usually produce a material that is not hot short.

The most common explanation for hot shortness in steel claims that iron and iron sulfide form a low melting eutectic which produces a liquid phase at the grain boundaries in the usual hot working temperature range. This grain boundary liquid phase embrittles the steel. Manganese combines with the sulfur to form a more refractory sulfide which avoids the liquid phase.

Certain facts are not entirely consistent with the common theory of hot shortness. The onset of hot shortness does not coincide with the temperature of the ironiron sulfide eutectic and there is apparently no discontinuity of properties at that temperature. Furthermore, the literature indicates that hot shortness is confined to a certain temperature range and that the steel is ductile above and below this range.

Recent careful examinations of steels indicate that the true phase equilibria are very complex and that simple iron sulfide or manganese sulfide phases do not exist in commercial materials. The sulfide inclusions in steels are not necessarily single phase. Thus, the solubility and behavior of the sulfides is variable and depends upon other elements that may be present in addition to iron, sulfur and manganese.

In this investigation an attempt was made to study the deformation of a number of laboratory-melted and commercial steels. A compressive load was applied to the steels at constant temperature and also during heating. The amounts and rates of deformation in these two types of tests was not consistent: in the constant temperature tests the hot short steels deformed more than the others while they appeared to deform less during heating. Metallographic examination of the sulfides indicated that the sulfides in cast steel are not stable and that they change during heating and hot working.

REVIEW OF LITERATURE

The lack of ductility in steels at elevated temperatures is called hot shortness and sometimes red shortness.

According to the Metals Handbook (1)* hot shortness is defined as "brittleness in hot metal" and red shortness is defined as "brittleness in steel when it is red hot". Thus, the term hot shortness can be used for all metals while the term red shortness should only be applied to steel. However, no consistent terminology is used in the literature of steel. The term hot shortness is used frequently and in this thesis it will be used to describe a condition of low ductility in steel at elevated temperatures that can be caused by sulfur.

Wohrman (2) reviewed some of the early ideas on hot shortness and presented his own theory which was entirely different from the generally accepted theory. Wohrman intimated that Le Chatelier in 1903 was one of the first to ascribe hot shortness in steel to the "melting of sulfide at about 980°C." Wohrman continued, "Today hot shortness due to sulfur is fairly universally explained by this tendency of the sulfide to form continuous envelopes around the 'grains' of the metal. The envelopes break up the

* Numbers in parenthesis refer to references in bibliography.

continuity of the metallic mass and cause, when melting at about 1795 degrees Fahr. (980 degrees Cent.), the falling apart of the metal. At still higher temperatures the molten sulfide is absorbed (dissolved) by the iron which then regains its continuity and plasticity." The absorption (dissolution) of the sulfide by austenite at very high temperatures was an idea credited to Ziegler (3).

Wohrman disagreed with the popular theory of hot shortness and proposed his own. He proposed that hot shortness was due to a natural lack of plasticity of austenite at the low end of its temperature range and that this low ductility was reduced to substantially zero by the solution of a small amount of sulfur. Furthermore, he felt that sufficient iron sulfide was soluble in austenite to cause hot shortness but that manganese sulfide was essentially insoluble. Since manganese effectively combined with the sulfur in the presence of iron, manganese prevented hot shortness by keeping the sulfur out of solution.

Wohrman cited the work of Sauveur and Lee (4) on "critical plasticity" to substantiate his theory. Sauveur and Lee had found that when a bar of steel was heated well into the austenite range at its center by a torch and then subjected to a twisting action, the steel did not deform at the center where the temperature was highest but, rather, it twisted at points equidistant from the center on either side. It was found that the steel bar twisted at the position where the microstructure was essentially all ferrite. This experiment convinced Wohrman that ferrite was plastic and that austenite was much less so. A slight reduction of the plasticity of the austenite by the solution of a little sulfur was all that was needed to cause hot shortness. Wohrman claimed that heating to very high temperatures caused the disappearance of hot shortness because the plasticity of the austenite increased with increase in temperature. Melting of sulfides was not involved in Wohrman's theory.

Howe (5) was one of the first to describe the return of ductility to a hot short steel at very high temperatures. He claimed that hot shortness could be circumvented by heating to a higher temperature and that manganese counteracted hot shortness at all temperatures and from all causes.

The fact that hot shortness was limited to a certain temperature range was difficult to explain. Wohrman explained it easily and simply. Ziegler's explanation was rather radical for his time. The solubility of sulfide in molten steel had been accepted for some time but sulfides were believed to be insoluble in solid steel. Solid solubility required proof.

The solubility of sulfides and other materials in liquid steels was believed by Sims and Lillieqvist (6), Benedicks and Lofquist (7) and others to be responsible for determining the size, shape, distribution and other characteristics of sulfides and other inclusions in steels. The nature of the inclusions, of course, determined their effects on the properties of the steel.

The deleterious effect of sulfur on the hot working characteristics of steels has been explained with the aid of the iron-sulfur phase equilibrium diagram shown in Figure 1. Figure 1 was given by Hansen and Anderko (8). The iron sulfide forms an eutectic with gamma iron at 988° C and 31 per cent sulfur (weight per cent). The solubility of sulfur in solid iron was determined by Rosenquist and Dunicz (9) and by Turkdogan, Ignatowicz and Pearson (10). The iron-rich side of this system is shown in Figure 2A and Figure 2B. The solubility in the gamma phase increases from 0.005 per cent sulfur at 913°C to 0.050 per cent sulfur at 1365° C. The invariant reaction at 1365° C is for the three phase equilibrium between gamma ferrite, delta ferrite and melt. The diagrams in Figure 2 show that liquid may form in iron-sulfur alloys at 988°C if the sulfur is sufficiently high. The formation of this liquid



Figure 1. Iron-Sulfur Equilibrium Diagram. (Hansen and Anderko)



Figure 2A. Iron-Sulfur Equilibrium Diagram. (Turkdogan, Ignatowicz and Pearson)



Figure 2B. Iron-Sulfur Equilibrium Diagram. (Turkdogan, Ignatowicz and Pearson)

phase has been the basis of the explanation for hot shortness in steels. It has been argued that a continuous film of liquid sulfide at the grain boundaries destroys the cohesion between the grains and consequently the material fails during hot working.

The solubility of sulfur in iron, Figure 2, would indicate that no liquid can form in steels with less than about 0.01 per cent sulfur. Thus, low sulfur material should not be subject to hot shortness according to the usual explanation for hot shortness. On the other hand, an alloy containing over 0.01 per cent sulfur may contain a small quantity of a liquid phase within the temperature range of 988° -1100°C.

Ainslie and Seybolt (11) do not quite agree with the phase diagram determined by Turkdogan, Ignatowicz and Pearson. They feel that the solubility of sulfur is 0.029 per cent at 900°C. There is general agreement that the iron-iron sulfide eutectic temperature is 988°C.

Hilty and Crafts (12) determined the iron-iron oxideiron sulfide diagram and found a ternary eutectic which melted at 925°C.

The minimum amount of sulfur which should not be subject to hot shortness is not known exactly. Figure 2 indicates that hot shortness should disappear below 0.01

per cent sulfur. However, some investigators (13) have reported that even when the sulfur content is as low as 0.008 per cent hot shortness can occur.

In a paper published by Joseffson, Koeneman and Lagerberg (14) hot shortness is related to the heat treatment of iron and steel. These investigators claimed that sulfur can cause hot shortness in steels by two different mechanisms. The first mechanism is a solid solution hardening effect. They showed that sulfur hardens and embrittles austenite, especially when the austenite is supersaturated with the sulfur, by showing that hot shortness was more pronounced in samples heated to a high temperature, 1050° - 1300° C, and then cooled to the testing temperature 850° - 950° C, than in samples heated directly to the testing temperature.

The second embrittling mechanism of sulfur proposed by Joseffson et al., involved the formation of thin grain boundary sulfide films. The effect of these films was demonstrated by quenching samples from 1300°C and then heating to 960°C where they were tested for hot shortness. The samples were hot short upon reaching 960°C and grain boundary sulfide films could be seen in the microstructure of these samples. Holding at 960°C for various times before testing showed that the hot shortness decreased with increased holding time and after 24 hours at 960° C the steel was no longer hot short. Metallographic examination revealed that the sulfide network spheroidized during holding at 960° C and that the degree of hot shortness was related to the degree of continuity of the grain boundary sulfides. Heating to 1150° C followed by furnace cooling to 960° C produced a very coarse grain boundary sulfide network. This network would not spheroidize at 960° C and the hot shortness of these samples did not decrease with holding time at 960° C.

Early work in torsional ductility studies was performed at elevated temperatures by Clark and Russ and also by Ihrig (15), (16) on iron base alloys in the gamma range. In this method the number of twists made before failure was taken as a measure of hot workability. Ihrig's results showed that even a small addition of sulfur decreased the number of twists before failure in the temperature range of $2100-2450^{\circ}F$ ($1150-1343^{\circ}C$). Steels with 0.029 per cent sulfur and 0.092 per cent sulfur showed 170 turns and 70 turns at $2100^{\circ}F$ ($1150^{\circ}C$), respectively. Clark claimed and emphasized that the rate of deformation was an important factor.

The significance of the torsion test results is questionable. The samples were about two feet long and

probably were not at a uniform temperature. Furthermore, a material that can survive 70 revolutions during torsion would appear to be ductile and the interpretation of the data is difficult. However, the torsion test method for measuring ductility has been revived by Rossard and Blain (17) and by Guenssier and Castro (18).

A recent study of iron-sulfur, iron-sulfur-oxygen and iron-sulfur-manganese alloys tested in tension in the temperature range 1600° to 2400° F at strain rates of about 0.001 to 100 per cent per second by Ogawa, King and Grant (19) revealed the following:

- "1. Ferritic iron alloys, with solid FeS inclusions, are more ductile at 1600°F than comparable austenitic alloys at 1700°F. The presence of the liquid FeS phase destroys all semblance of ductility even in the presence of small amounts of oxygen, which tends to globularize the inclusions. Solution of the liquid iron sulfide in gamma iron restores some ductility at high temperatures, but the ductility is still greatly impaired by increasing sulfur content.
 - In view of the poor ductility, strain rate effects are not very significant in the straight Fe-S alloys.

- 3. The effect of strain rate becomes more evident in the more ductile Fe-S-O and Fe-Mn-S alloys, the ductility being greatest, apparently, at intermediate strain rates.
- 4. Both oxygen and manganese additions effectively spheroidize the sulfide inclusions; ductility is largely regained in proportion to the amount of oxygen and manganese added. The oxysulfides tend to be coarser than the (Mn, Fe)S inclusions. High Mn:S ratios (above 20) at high manganese contents decrease ductility, even at 2400°F, through formation of a two phase eutectic-type structure."

The effect of manganese on the solubility of sulfur in solid iron was determined by Turkdogan and Ignatowicz (10). Figure 3 shows their data for the manganese and sulfur contents of solid solutions in iron in equilibrium with manganese sulfide for 1200° and 1335°C. The low values of about 0.004 per cent at 1200°C in Figure 3 may be compared with the solubility of sulfur in pure iron which is shown to be 0.031 per cent at this temperature in Figure 2B.

According to Gain(20) manganese may prevent hot shortness in iron when present to the extent of three times the sulfur percentage if the oxygen percentage is not above





0.04. The minimum amount of manganese needed to eliminate hot shortness in steels of various sulfur contents is not known.

Oxygen alone should not cause any hot shortness. Gain said that oxygen in amounts up to 0.20 per cent does not cause hot shortness in pure iron if sulfur is below 0.01 per cent, but the presence of considerable amounts of oxygen in irons (0.1 per cent and above) tends to reduce the efficiency of manganese in preventing hot shortness because the manganese tends to be present as oxide rather than sulfide.

Josefsson, Koeneman and Lagerberg (14) did not find oxygen to cause hot shortness and they said that there seemed to be no basis for assuming oxygen to cause hot shortness because the oxygen solubility in gamma iron even at high temperatures appears to be very small.

The effect of copper content on hot shortness of iron and steel has been argued by many investigators for a long time. No decisive conclusion has been reached. The ironcopper phase diagram in Figure 4 shows that hot shortness should not occur in ordinary iron and steel because no liquid phase can form in the normal hot short temperature range. The equilibrium diagram shows that alloys containing over 7.5 atomic per cent (or about 8 weight per cent)



Figure 4. Iron-Copper Equilibrium Diagram (Daniloff)

of copper can be partially liquid when heated above 1094°C. The molten copper-rich constituent would tend to be formed at the grain boundaries and would cause hot shortness if hot working were attempted at such temperatures. Ordinary steels do not contain a sufficient amount of copper to form any liquid phase. However, if the copper became segregated, it might become possible to form a liquid phase at the grain boundaries at 1094°C and above.

EXPERIMENTAL

A. Tester:

The compression tester used in this study is shown in the drawing of Figure 5A, while Figure 5B is a photograph of the apparatus.

Load was transmitted to the sample by two pieces of Type 304 stainless steel. The nature of the holes at the ends of these bars for holding the sample is shown in the drawings of Figure 6. The 3/16 in. groove on the side of the upper bar and the 3/16 in. diagonal hole permitted the placing of a thermocouple inside the sample. A chromelalumel thermocouple was used in this manner to measure the temperature of the samples.

The tube type furnace was 18 inches long and was constructed for this study. Kanthal heating element wire was wrapped onto an alundum furnace tube. This was placed inside another ceramic tube which was surrounded by insulating bricks. The outer shell was transite.

Temperature control on the furnace was obtained with a second chromel-alumel thermocouple whose hot junction was located between the two tubes, immediately adjacent to the heating element. This control thermocouple and Leeds and Northrup controller activated a relay in a simple on-off control circuit.



Figure 5A. Apparatus For Applying Static Compressive Load To Samples



Figure 5B. Apparatus For Applying Static Compressive Load To Samples



Figure 6. Stainless Steel Bars For Holding and Loading Samples The location of the hot junction of the control thermocouple gave a rapid response to temperature change and the thermal lag produced by the furnace tube gave a relatively uniform temperature in the sample for most of the constant temperature experiments. The portable potentiometer used for measuring the output of the sample thermocouple gave a constant reading in these experiments. In several experiments, however, for some unknown reason the sample temperature cycled up and down in a temperature interval of approximately 10 degrees Centigrade.

Load was applied to the sample by means of a lever system. Two 2-inch angle posts were welded to the right end of the 9-inch channel which served as the base, see Figure 5A and Figure 5B. The lever arm was a 3-inch channel which was pivoted by a pin at the top of the posts. A hanger at the left end of the lever arm was provided to hold lead bricks which were used as the load. The lead bricks weighted 26 1/8 lb each. The geometry and weight of lever arm was such that a force of 420 lb was applied to the sample with one brick on the hanger. This load gave a stress of 2100 psi in a 1/2 in. diameter sample.

The change in length was determined by means of a pointer attached to the lever arm and a scale attached to one of the l-inch angle iron posts which served as guides

for the lever arm. Gentle tapping of the lever arm prior to each reading avoided erratic readings due to sticking of the arm. The smallest division on the scale was 1/32 in. and the location of the scale was such that the length change of the sample was multiplied by a factor of 5.4. The length change was for both stainless steel bars and sample. It was assumed that the stainless steel bars did not deform measurably with the loads being used. However, the thermal expansion of the stainless steel bars caused a large part of the length change when the temperature changed. The thermal expansion of the stainless steel bars was assumed to be the same for each experiment. Length change was measured every 1/2 min. in the constant temperature experiments and every 1 min. in the heating experiments.

B. Preparation of Specimen:

Three commercial steels and several experimental steels were studied. The experimental heats were made by Yen (21). Yen melted the steels in an Ajax high frequency induction furnace. The nominal compositions of all the steels are listed in Table 1.

Two different sample sizes were used. The larger had a diameter of 1/2 in. and length of 1 in. The other had a

TABLE 1

COMPOSITION OF SPECIMENS

Specimen Number	С	Si	Р	S	Mn	Cu	A 1
0				0.16	0.0/		
H3	0.48	0.94	0.003	0,46	0.84	•••	0.06
H4	0.40	0.25	-	0.10		1.50	0.10
н5	0,40	0,25	-	0.10	1.00	1.50	0.10
Н6	0.40	0.25	-	0,30	1.00	1.50	0.10
H7	0,40	0.25	-	0.10	-		0.10
н8	0.40	0.25	-	0,05	-	-	0.05
H11	0,40	0.25	-	0,10	1.50	1509	
AISI							
1018	0.15-0.20	-	0,04	0,05	0.60-0,90		-
AISI					·		
1042	0,44-0,52	-	0,04	0.05	1.10-1.40	-	
Ingot Iro	n – n	-	-		-	-	-

diameter of 3/8 in. and a length of 1 in. All the samples were machined on a lathe. One end of each specimen was drilled axially with a 3/16 inch drill to a depth of 1/2inch in order to provide a hole for the thermocouple.

C. Test Procedure:

The first step in making a compression test was to place the sample in the furnace. The sample was placed inside the hole in the lower stainless steel bar with the hole in the sample facing up. The thermocouple protruding from the upper stainless steel bar was inserted into the hole in the sample and the upper end of the sample went into the hole in the top stainless steel bar.

The lever arm was then put into position. Several pieces of soft copper and lead sheet were placed between the stainless steel bar and the lever arm in order to avoid point contact. The stainless steel bars and the sample were lined up as carefully as possible to give axial loading. With the sample and lever arm in place the apparatus was ready for the start of an experiment. For a constant temperature test the power was turned on and the furnace heated to the test temperature. The temperature of the sample was watched and when it became constant, the required number, usually one, of the lead bricks was put on the hanger at the end of the lever arm. The temperature of the sample and the position of the lever arm were determined every 1/2 min. Figures 7 and 9 show data for this type experiment.

At the end of a test the load was immediately removed from the sample. If the sample deformed too much it became very tightly wedged in the holes in the stainless steel bars and it was extremely difficult to remove. Usually the stainless steel bar was pulled up from the furnace and the sample was removed as soon as possible.

In the heating tests, the lead bricks were put on the hanger and the power turned on. The controller was set to limit the temperature rise of the furnace. The temperature of the sample and the position of the lever arm were determined every 1 min. until the highest temperature of the experiment was reached at which time the load was removed and the sample was taken out of the furnace.

Some of the difficulties encountered in the work have been mentioned above. Others were:

The controller functioned well most of the time but occasionally it would not hold a constant temperature and the temperature of the sample fluctuated up and down in an approximately 10 degrees Centigrade temperature range. Another annoying problem was caused by the furnace temperature changing from one experiment to the next. In the constant temperature runs it was found that the controller had to be adjusted for each experiment in order to have the sample at the correct temperature; the furnace would not bring the new sample to the same temperature as the last. This adjustment of sample temperature was time consuming and required exposing the sample and stainless steel bars to the high temperature for extra periods of time. It required approximately 1 hr. to obtain the correct constant temperature.

The difficulty with the temperature control was particularly annoying because the stainless steel bars were not sufficiently heat resistant for the temperatures being used. Oxidation of the bars and sample were partly responsible for difficulty in removing the samples and it also limited the life of the bars. Another problem involved buckling of the bars which cracked the furnace tube on several occasions. It was necessary to straighten the stainless steel bars periodically during the investigation.

RESULTS AND DISCUSSION

A. Compression Testing:

According to the commonly proposed theory for hot shortness in steel, sulfur produces a liquid phase at the grain boundaries which destroys the cohesion between grains and allows the steel to fall apart. If this theory was correct the original experiments in this investigation should have revealed the lower limit of the hot shortness range. These experiments applied a static compressive load to the sample during heating. Upon entering the hot shortness range with the strength and ductility markedly reduced, the lever arm on the tester should have dropped or, at least, indicated some change in deformation rate. This did not happen.

One of the first samples tested was an as-cast sample from the experimental steel H8. This steel contained no manganese and was known to be hot short. Yen (21) had attempted to forge this steel and had found it to be hot short. A somewhat sudden yielding of the sample upon entering the hot shortness temperature range was anticipated but was not found.

The length change for the stainless steel bars and the H8 sample during heating to 1100°C is shown in Figure 7.


Since the strength of the steel in the vicinity of the hot shortness range was not known, six lead bricks giving a stress of 9350 psi. were used in this experiment. This stress caused the sample to start deforming at an appreciable rate at about 650° C. The deformation was indicated by a decrease in length, dropping of the beam, which continued over a temperature range up to about 800° C. The stationary range of the beam between 800° C and 900° C is probably due to the alpha and gamma iron existed together and apparently the gamma iron is stronger than alpha iron.

After the equipment cooled down, an attempt was made to examine the sample. The sample was found to be upset in the holes at the ends of the stainless steel bars and was removed with great difficulty. No evidence of failure could be found in this sample. Apparently the sample had deformed below the hot shortness range and the length change above 900°C was due to thermal expansion of the stainless steel bar.

The supply of the experimental steels with no manganese was limited and further work to determine the proper stress level was done with ingot iron samples. The samples were heated to $1000-1100^{\circ}$ C with no load and then various loads were applied to determine the load that would cause a slow deformation at the testing temperature. The early experiments indicated that when the samples were allowed to deform as much as conditions would permit, their removal from the stainless steel bars after the experiment was a problem. For this reason the amount of deformation of samples was restricted by removing the load before the sample became tightly wedged in the holes in the stainless steel bars.

Figure 8 shows the results for an ingot iron sample in a static load, constant temperature run. The length change as a function of time is shown for two different stress levels. After the sample reached 1100°C and the lever arm reached a stable and constant position one lead brick was placed on the hanger to give 2100 psi in the sample. The sample showed a rapid initial deformation. Eight minutes after loading the sample stopped deforming. At eleven minutes the temperature began to drop and finally became constant at 1030°C. A second lead brick was added 26 minutes after the first. This higher stress of 3460 psi caused another short period of rapid deformation followed by a much reduced deformation rate.

Experiments such as that described above indicated that a load of one lead brick, giving a stress of 2100 psi on a 1/2 in. diameter sample, was sufficient to deform the steel in the 1000-1100^oC temperature range. The stress



produced by the weight of the lever arm alone did not produce a measurable amount of deformation in the time involved in these experiments, Figure 9. The weight of the lever arm and hanger produced a stress of 635 psi. in a 1/2 in. diameter sample.

Figure 9 shows data for an experiment with a sample of H8 steel. This was the constant temperature, static load test for which the data are shown in different form in Figure 10. The lever arm with no bricks on the hanger was applying load to the sample during heating. In the vicinity of 700°C slight irregularities in the heating and length change curves of Figure 9 are due to phase transformation in the sample. In this experiment no difficulty was experienced in bringing the sample to the proper constant temperature. The temperature became constant quickly and the thermal expansion of the bar and sample stopped as indicated by a constant value for the length. No measurable length change occurred after the sample temperature became constant until the stress was raised by putting a lead brick on the hanger at the end of the lever arm 57 min. after the start of the experiment. A rather large contraction of the sample during the first one-half minute of loading was measured and then further deformation occurred much more slowly. The data in Figure 9 are typical of the



constant temperature, static load tests described below except for the fact that much more difficulty was frequently encountered in bringing the sample to a constant temperature of 1100° C.

A series of experiments was run at constant temperature and constant static load to determine any difference in deformation behavior of steels known to be hot short and those not hot short. The results of these experiments are shown in Figure 10. A static load of 2100 psi. was used and the temperature was 1100°C for all samples except H7 which was 1150°C. Steels H4, H7 and H8 had been found to be hot short by Yen (21). The AISI 1018 and 1042 steels and the ingot iron were commercial wrought materials which were not hot short. Steel H11 was an experimental steel on which no attempt at hot working had been made but this steel contained manganese and was probably not hot short.

The results of the constant temperature tests appeared to separate the steels into two groups on the basis of deformation rate. The hot short steels were in one group while the steels that were not hot short were in the other group. The hot short steels were experimental heats to which manganese had not been added. The other steels contained manganese.

Figure 10 shows that both the initial rapid deforma-



tion rate and the subsequent slower rate for the hot short steels are greater than for the steels not hot short. The initial rapid deformation occurred during the first half minute. After one half minute of load application, the deformation of the hot short steels was greater in each case than that of the other steels. This deformation is given in column two of Table 2. The deformation at the end of six minutes is given in the third column and the last column in Table 2 gives the average rate of deformation during the period between one half and six minutes. The data show that after the first half minute rapid deformation period the rate of deformation was still more rapid in the hot short steels.

The data for 1018, 1042 and ingot iron indicated relatively similar behavior for these materials. The average rate of deformation during the first half minute was approximately 0.01 in./in. Their deformation rate then became approximately 0.005-0.006 in./in. The deformation rates are slightly different toward the end of the tests and are not in the expected relative order. The deformation rate of the 1042 steel was the greatest while that of the ingot iron was the least. Since the 1042 steel with its higher carbon content is the strongest of the three materials it seems reasonable to expect it to offer the

TABLE 2

AVERAGE DEFORMATION RATE FOR CONSTANT TEMPERATURE EXPERIMENT (SLOW DEFORMATION PERIOD)

Specimen Number	Deformation at 1/2 min. in/in.	Deformation at 6 min. in/in.	Average Deformation Rate in/in/min.
	0.0720	0 1/19	0.0127
HO	0.0720	0.1100	0.0127
H4	0.0695	0,1180	0.0093
H7	0.0520	0.0965	0.0081
H11	0.0374	0.0573	0,0036
AISI			
1042	0.0114	0.0460	0.0063
AISI			
1018	0.0089	0.0407	0,0057
Ingot Iron	n 0.0108	0.0359	0.0046

most resistance to deformation. This unexpected order of deformation rates could probably be due to factors other than carbon content, such as grain size which tends to give more grain boundary area which should increase the viscous flow.

The hot working characteristics of the Hll steel were not known but the composition of this steel was such that it was not believed to be hot short. There was some doubt about the validity of the test data on this steel. The initial rapid deformation rate was high for a steel not susceptible to hot shortness. The rapid deformation rate of H11 was intermediate between the hot short group and the not hot short group. The subsequent slow deformation rate for H11 was not very constant as shown in Figure 10. The data showed that the temperature did not remain constant during the experiment. The controller did not function properly and allowed the sample temperature to fluctuate. While the temperature was increasing (from approximately 1095°C to 1105°C) the curve showed a slow deformation rate and while the temperature was decreasing (in approximately the same temperature range) the curve showed a more rapid deformation rate. During heating, thermal expansion would counteract the deformation from the compressive load while the thermal contraction during cooling would increase

the apparent deformation. The low deformation rate during the later stages of the test does not appear to be significant. The reason for the relatively high initial deformation rate is not known.

Experimental steel H7 apparently picked up some manganese from the crucible in which it was melted even though none was added intentionally (21). This steel behaved much like the other two hot short steels but showed lower deformation rates which could have been the result of the small amount of manganese present in this steel. However, this could have been the result of experimental error. The difference in deformation rates between H7 steel and the other two hot short steels is the expected difference if hot short steels deform more rapidly in this test and if the manganese in H7 decreased its degree of hot shortness slightly.

All the experimental steels tested in the constant temperature series of experiments were in the as-cast condition. The samples were machined from the ingots. The initial rapid deformation rate of these experimental steels was higher than that of the commercial wrought materials. The difference in deformation rate could have been due to differences between cast and wrought metals. Static load, constant temperature tests were performed on

both cast and wrought samples of three different experimental steels in order to determine if the differences in deformation rates shown in Figure 10 were due to hot shortness in some of the steels or if it was a difference between cast and wrought steels. The results of this series of tests is shown in Figure 11.

The steels used in the cast versus wrought metal tests were those which Yen (21) had worked into 3/8 in. rods. These were his H3, H5 and H6 steels. Samples were cut from the rods and from the ingots of these steels. They were all 3/8 in. diameter. The same load was used in these experiments as was used in the tests whose results are shown in Figure 10. The smaller diameter of the samples in this series resulted in a higher stress of 2800 psi.

The results of the tests on the cast and the wrought samples presented in Figure 11 show that the amount of deformation during the first half minute of load application was approximately the same for all the samples, 0.05-0.06 in./in. The subsequent deformation rate varied considerably and indicated no consistent variation with condition of the metal, cast or wrought. The deformation of the H3 samples after the first half minute was erratic. The cast sample from H6 steel deformed more rapidly than the wrought sample while the reverse was true for the H5



steel. Since there was no consistent and definite difference in deformation rate during the slow deformation period in the later part of the tests and since the initial rapid deformation rate was the same for both cast and wrought samples it would appear that cast and wrought samples behave in a similar manner in the static load, constant temperature tests. The test results shown in Figure 11 indicate that the difference between the H4, H7 and H8 steels compared to the H11, 1042, 1018 and ingot iron shown in Figure 10 is not the difference between cast and wrought metal. The difference between the two groups of samples in Figure 10, if it is significant, could be a difference between hot short steels and steels that are not hot short.

The results of the heating experiments are shown in Figures 12 and 13. The curves are all very close together and they are presented in two figures in order to separate them. All the samples in these experiments had a diameter of 1/2 in. and they were all heated while stressed to 2100 psi.

In Figures 12 and 13 "expansion" which is the ordinate is actually the total increase in length of the stainless steel bars and the sample divided by the original length of the sample. The total increase in length was due to





thermal expansion of bars and sample less any contraction of the sample due to deformation by the compressive load.

The data for the H8 curve in Figure 12 was obtained from the data shown in Figure 9. This sample was actually loaded with the weight of the lever arm but this was not sufficient load to cause deformation. Figure 9 shows that with this light load no length change occurred in the sample as long as the temperature remained constant. The H8 sample with the lever system expanded a total of 29.9 in./in. of sample upon heating to 1080°C. Comparison of the H8 curve with the other curves shows that the 2100 psi stress was sufficient to deform the austenite during heating.

The five steels heated with a constant static stress of 2100 psi were H4, H7, H11, 1018 and 1042. Steels H4 and H7 had not survived hot forging and were known to be hot short. The 1018 and 1042 were commercial wrought steels and could not have been hot short. Steel H11 contained sufficient manganese to prevent hot shortness but had not been hot worked. Thus, H4 and H7 were hot short while H11, 1018 and 1042 were not.

All five steels gave very similar curves for temperatures below the critical range. Their behavior in the critical range was somewhat erratic. Above the critical range where they became austenitic two differences could be

detected between the two types of steel, the hot short steels and those that were not hot short. First, the curves for the hot short steels were steeper than the others. This can be seen in Figure 12 where the curves for H4 and H7 above about 750°C are approximately parallel and steeper than the curve for H11. Comparison of the slopes of the curves for H11, 1018 and 1042 in Figure 13 shows that they too are approximately parallel.

The second difference was in the maximum of the curve. The two hot short steels expanded more and reached higher temperatures before the rate of deformation of the sample under the compressive load exceeded the rate of thermal expansion of the stainless steel bars and the sample. The hot short steels H4 and H7 had maxima at 1125°-1150°C. H11 steel had a maximum at about 1110°C while the two commercial wrought steels had maxima around 1070°C.

These results from the heating experiments agree with the theory advanced by Joseffson et al. (14) and, on the other hand, they do not appear to agree with the results Of the constant temperature tests whose results are shown in Figure 10. These peculiarities of this data are discussed below.

Joseffson and his co-workers claimed that sulfur causes hot shortness in steels because of its effect on the

properties of austenite. To some extent, this is similar to the theory proposed by Wohrman (2). Dissolved sulfur lowers the ductility and raises the strength of austenite. The addition of manganese to steels is effective in the elimination of hot shortness because it preferentially combines with the sulfur forming a sulfide that is practically insoluble. When manganese is not present the sulfur in the form of iron sulfide dissolves sufficiently to affect the properties of the austenite adversely.

The three curves for the stressed samples in Figure 12 show that the steel with the least manganese had the highest resistance to deformation. and the steel with the highest manganese deformed most at temperatures in the austenite range. The curve for steel H4 had the highest maximum which indicated the least deformation and this steel contained no manganese. The H11 steel had 1.5 per cent manganese and had the lowest maximum. It is interesting to note that metallographic examination of heated samples of the H7 steel indicated that it contained a small amount of manganese, probably inadvertently added to the steel from the slag of a previous heat remaining in the crucible in which it was melted (21). The H7 steel with somewhat higher manganese showed a lower maximum than H4 steel.

If the results from the heating experiments are the

expected results based on the effect of dissolved sulfur on the properties of austenite, the results of the constant temperature experiments shown in Figure 10 appear to be the reverse of the expected results. The curves in Figure 10 show that the hot short steels, those which should have the highest concentration of dissolved sulfur, deformed the most under the compressive load at constant temperature. It seems reasonable to expect the same deformation process to operate at constant temperature as the process operating during relatively slow heating. This would mean the hot short steels should either deform the most or the least in both sets of emperiments. No explanation can be offered for the different behavior of the hot short steels in these experiments.

Yen's (21) experience with the experimental steels studied during this investigation indicated that manganese is very effective in eliminating hot shortness. Steels to which no manganese was added were found by Yen to be hot short while those to which some manganese was added were not hot short. Steel H8 with only 0.05 per cent sulfur and with no manganese was hot short. Steel H3 with 0.46 per cent sulfur and only 0.84 per cent manganese was not hot short. A small amount of manganese can apparently overcome the

adverse effect of very high sulfur.

Ogawa, King and Grant (19) found pure iron-sulfur alloys to be insensitive to strain rate with tensile loads. The addition of oxygen and manganese increased sensitivity to strain rate giving highest ductility at intermediate rates, 0.1-1.0 per cent elongation per second. In this investigation no evidence of failure from the static compressive loads was found. Steels that failed quickly from the impact loading of a forging hammer flowed readily in these tests. These steels were sensitive to strain rate under compressive load and the results suggest that sensitivity to strain rate may vary considerably for tensile and compressive loads.

B. Metallographic Examination:

The sulfides were examined metallographically in an attempt to gain additional information on the hot shortness phenomenon.

Metallographic specimens were taken from the tested samples and from the bar stock and ingots from which the samples were made. Polishing was similar to the procedure used by Yen (21). The final polishing was done on Microcloth or silk using Linde B polishing compound. Examination and photomicrography were done on a Bausch & Lomb Research Metallograph. Metallographic examination indicated that the nature and distribution of the sulfides in the cast steels were not stable and they changed when the steel was hot worked or heated to the temperatures used in these experiments. The sulfides in the wrought steels showed very little change as a result of the heating and deformation in these experiments. The presence of iron sulfide in a cast steel does not indicate a susceptibility to hot shortness but iron sulfide in a steel that has been heated for a long time at a sufficiently high temperature is probably an indication of a degree of hot shortness. Certain sulfide shapes and distributions may also indicate hot shortness in heated steels.

Typical photomicrographs of the sulfide inclusions in the steels are shown in Figures 14-36.

The samples from the H3, H5 and H6 steels showed that there was an appreciable change in the sulfides when the ingots were worked into bars, Figures 14, 16, 18, 20, 22 and 24. The sulfides in the bars were elongated in the longitudinal direction indicating that the sulfides were plastic at hot working temperatures. Steels H5 and H6 showed clusters of sulfides in their ingots, Figures 18 and 22, which were eliminated by hot working and they were not found in the bars wrought from these steels, Figures

20 and 24. Examination of the various specimens indicated a tendency for the sulfides to be smaller in the wrought bars than in the ingots from which the bars were made. This effect was noticeable in the very high sulfur steel, H3, which had very large sulfides in the ingot, compare Figures 14 and 16. Duplex sulfide inclusions, inclusions which appeared to contain two phases, iron sulfide and manganese sulfide, were found in the ingot of steel H3. See inclusions which are marked "D" in Figure 14. No duplex sulfides were found in the H3 bar or in any samples of this steel that had been heated to 1100°C. After heating or hot working, the sulfides in H3 steel were all manganese sulfide. Iron sulfide could be differentiated from manganese sulfide by its color and because it was optically anisotropic in polarized light. The anisotropic iron sulfide became alternately bright and dark when it was viewed through crossed nicol prisms with polarized light while it was being rotated with the stage of the microscope. The optically isotropic manganese sulfide remained dark in all positions when examined under these conditions.

Heating samples machined from the ingots to approximately 1100^OC as was done in most of the experiments in this investigation caused some changes in the sulfide inclusions. There was a tendency for the sulfides to coarsen. This is pronounced in steel H3, Figures 14 and 15, and is also noticeable in many of the other experimental heats. In a steel like H4 in which the as-cast sulfides tended to be grain boundary film-like inclusions, Figure 28, the heating and slight deformation during the test caused the long film-like inclusions to break up.

In the hot short steels, H4, H7 and H8 practically all of the sulfides in the ingots were iron sulfide which, in H4 and H7, had a tendency to be film-like, Figures 28 and The sulfides in H4 and H8 were still iron sulfide in 30. the samples after testing. In H7, which contained a small amount of manganese, most of the sulfides after testing were manganese sulfide and only a small amount of iron sulfide could be found in the tested sample. The hot short steels displayed some sulfides with a triangular shape after testing, Figures 29 and 31. These triangular inclusions were almost all iron sulfide. It is believed that these inclusions were located at the junctions of grain boundaries and that they had been liquid at high tempera-Since the iron-iron sulfide eutectic can melt at tures. 988°C, liquid could have been present in the hot short steels H4, H7 and H8 at the temperatures used in the tests. Other sulfide inclusions in these steels appeared to be located in a grain boundary pattern.

Metallographic examination of these samples created the impression that the theory of hot shortness which postulated liquid iron sulfide at the grain boundaries was the correct theory. In the hot short steels iron sulfide tended to stay at or move to austenite grain boundaries and the shape of these inclusions indicated that they could have been molten at the elevated temperature. Manganese sulfides which have a higher melting point than iron sulfides appeared to be more or less randomly distributed in the steels that were not hot short.

The film-like grain boundary sulfides in steel H4 were occasionally found to have a blue or brown color. These colors may have been produced by the 1.5 per cent copper present in this steel. H4 had no manganese and was hot short while the H5 and H6 steels were not. H5 and H6 steels had 1.5 per cent copper and 1.0 per cent manganese. H5 had 0.1 per cent sulfur while H6 had 0.3 per cent compared to 0.1 per cent sulfur for H4. It would appear that copper may be present in the sulfide phase of a steel but that copper will not prevent hot shortness.

The commercial, wrought steels AISI 1018 and AISI 1042 and the ingot iron did not show a noticeable change in their inclusions during testing in these experiments. There was nothing unusual about the inclusions in these materials.



Figure 14. Sulfide Inclusions In H3 Steel Ingot. Unetched, 500X



Figure 15. Sulfide Inclusions In H3 Steel Ingot Sample After Test. Unetched, 500X



Figure 16. Sulfide Inclusions In H3 Steel Wrought 3/8 Inch Bar. Unetched, 500X



Figure 17. Sulfide Inclusions In H3 Steel Wrought Sample After Test. Unetched, 500X



Figure 18. Sulfide Inclusions In H5 Steel Ingot. Unetched, 250X



Figure 19. Sulfide Inclusion In H5 Steel Ingot Sample After Test. Unetched, 250X



Figure 20. Sulfide Inclusions In H5 Steel Wrought 3/8 Inch Bar. Unetched, 250X



Figure 21. Sulfide Inclusions In H5 Steel Wrought Sample After Test. Unetched, 250X



Figure 22. Sulfide Inclusions In H6 Steel Ingot. Unetched, 250X



Figure 23. Sulfide Inclusions In H6 Steel Ingot Sample After Test. Unetched, 250X



Figure 24. Sulfide Inclusions In H6 Steel Wrought 3/8 Inch Bar. Unetched, 250X



Figure 25. Sulfide Inclusions In H6 Steel Wrought Sample After Test. Unetched, 250X



Figure 26. Sulfide Inclusions In Hll Steel Ingot. Unetched, 250X



Figure 27. Sulfide Inclusions In Hll Steel Ingot Sample After Test. Unetched, 250X



Figure 28. Sulfide Inclusions In H4 Steel Ingot. Unetched, 500X



Figure 29. Sulfide Inclusions In H4 Steel Ingot Sample After Test. Unetched, 500X



Figure 30. Sulfide Inclusions In H7 Steel Ingot. Unetched, 250X



Figure 31. Sulfide Inclusions In H7 Steel Ingot Sample After Test. Unetched, 500X



Figure 32. Sulfide Inclusions In H8 Steel Ingot. Unetched, 500X



Figure 33. Sulfide Inclusions In H8 Steel Ingot Sample After Test. Unetched, 500X


Figure 34. Inclusions In Ingot Iron Wrought Bar. Unetched, 250X



Figure 35. Inclusions In 1018 Steel Wrought Bar. Unetched, 250X



Figure 36. Inclusions In 1042 Steel Wrought Bar. Unetched, 250X

CONCLUSIONS

- Hot short steels do not fail during slow deformation by compressive load.
- 2. The deformation characteristics of the hot short steels were found to be somewhat erratic. During heating with a static compressive load applied, the hot short steels deformed less than the steels which were not hot short. The hot short steels deformed more than the others in the constant temperature experiments.
- 3. No sudden loss of strength by hot short steels was found when these steels entered their hot short range during heating under a static compressive load that would cause only slow deformation. In fact, no indication of hot shortness was found under these conditions.
- 4. The hot short range, if it exists, has not yet been determined experimentally. If melting of an eutectic containing iron sulfide is the cause of hot shortness, the hot short range may be a limited temperature range for low sulfur steels, perhaps up to about 0.05 per cent sulfur, where the sulfur present can be dissolved at high temperatures. However, in higher sulfur materials the liquid phase cannot be eliminated above

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the eutectic temperature and it does not appear reasonable to expect hot shortness to disappear at high temperatures in such materials.

- 5. Appreciable amounts of iron sulfide can be present in an as-cast steel even when manganese is present. The presence of much sulfur and some iron sulfide in an as-cast steel does not mean the steel is hot short. The presence of sufficient manganese can eliminate hot shortness when sulfur is as high as 0.5 per cent and perhaps when it is higher.
- 6. Steels that are not hot short will not contain any iron sulfide after they have been heated to sufficiently high temperatures for sufficiently long times and/or been hot worked. The time-temperature relationship for eliminating iron sulfide from cast steel was not studied. However, the presence of iron sulfide in a steel after prolonged heating at high temperature probably indicates that the steel is susceptible to hot shortness. The presence of grain boundary networks of sulfides and triangular-shaped sulfides at the junctions of grain boundaries in heated steels also probably indicates a susceptibility to hot shortness.

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VITA

Koang-Hwa Kung was born in Peiping, China on September 14, 1934. His native place is Manchuria according to Chinese custom. He received his primary education in Chungking, the war time capital of China during the Sino-Japanese war, and completed his high school in Taiwan (Formosa) in 1954. He then entered Taiwan Provincial Cheng-Kung University and received the Bachelor of Science degree in Metallurgical Engineering in 1958. After graduating from Cheng-Kung University, he served in Chinese Marine Corps for 20 Months. Discharged from the Chinese Marine Corps, he has worked in Taiwan Aluminum Corporation for almost three years. He then came to the United States and enrolled as a graduate student in Metallurgical Engineering at the University of Missouri at Rolla in September, 1963.

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