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MONTANA SCRIPTING STATES CHROMIZING OF STEELS

EDWARD BUCHAMAN

BY ...

A Thesis

Submitted to the Department of Metallurgy in Partial Fulfillment of the Requirements for the Degree of Bachelor of Science in Metallurgical Engineering

> Montana School of Mines Butte, Montana

> > May 17, 1949

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INTRODUCTION

In recent years, considerable thought and study have been given to the use of chromized articles in place of chromium stainless steel articles. The present extensive application of chromizing, indeed, helps greatly to conserve this valuable metal, as the quantity of chromium required for an ordinary case, about 0.1 mm. $(.004"$ thick), is only 0.2 Kg/m² $(.041.1b/ft^2).$ ¹ It is desirable, although chromizing supplies are limited, to use this metal for surface alloying, not only because of its excellent resistance to corrosion under a variety of severe conditions. but also because of its ability to undergo flanging and bending operations without spalling.

The process of surface alloying, or chromizing, is made possible through the phenomenon of diffusion of chromium atoms into the lattice of alpha iron. Before this phenomenon of diffusion is discussed, it is advisable to recall certain fundamental facts about the structure of the crystal lattice.

The ideal lattice structure consists of a three-dimensional array of atoms, each of which is held in its lattice position by forces that are identical with those acting upon all other

identical atoms. These atoms are not fixed in their respective sites but, as a result of thermal energy, they oscillate about these mean positions. The oscillations are synchronous, since each atom is bound to its neighbor. The frequencies of the modes of vibrations in a solid are limited by the boundary conditions and by the total energy.

A consideration of a static lattice (that is, one that is frozen or immobile as in Figure 1)² will indicate that all atoms will occupy positions at the bottom of the well. Under normal conditions the kinetic energy of an atom is much less than ϵ , ϵ _r, the potential energy of the well. Occasionally, however, an atom may acquire, from an outside source, a kinetic energy equal to or greater than \mathcal{E}_1 - \mathcal{E}_2 . Atoms can be raised from the normal state to higher energy states by bombarding them with electrons, or by subjecting them to high temperatures, or by allowing them to absorb radiant energy from an external source. The atom in one of the

higher energy states is said to be in an excited state (c_i) . When the energy absorbed by an atom is greater than ϵ - ϵ , then the atom is loosened from its atomic position, and it no longer constitutes an integral part of the elastic spectrum.

The phenomenon of diffusion depends on the loosening of the atoms in both the solvent and the solute. Therefore, when the energy absorbed by the atoms is greater than ε - ε they rupture their respective bonds and in consequence leave a vacant site to be filled by another atom. This, then, is the process that makes diffusion possible within the lattice.

It would seem that once an atom has acquired sufficient kinetic energy to leave its well, it would be independent of time. In general this is not true. Because of steric effects, atoms with sufficient kinetic energy to be loosened may, on approaching the upper walls of the potential energy well, collide with a neighboring atom and thus rebound into their well. Therefore, loosening may not occur with every attempt. Hence the time required for loosening depends upon both the geometry and size of the atom and its neighbors, and upon the period of atomic oscillation, temperature being of little influence.

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According to Mehl³, in order to have diffusion within the grain, the diffusing atoms must form a solid solution. In other words, before diffusion can take place, the solute atomic species have to be soluble in the lattice of the solvent species. In so much as there are two types of solid solution, there must be two corresponding types of diffusion, mamely, interstitial and substitutional diffusion.

In the former case a good example is the diffusion of carbon into gamma iron. The largest "holes" in the lattice of gamma iron are at the points of $\frac{1}{2}$ 0 0 and $\frac{1}{2}$ $\frac{1}{2}$, and here the small carbon atom is able to fit in with only a slight expansion of the unit cell. When a solvent lattice accepts an atom interstitially, there is always an expansion. Since the diffusion of carbon into gamma iron forms a type of solid solution different from that of pure gamma iron, any addition of a foreign atomic species would have to be effected by substitutional diffusion. It is this latter type of diffusion which will be studied herewith.

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DIFFUSION IN SOLID SOLUTIONS

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In a substitutional solid solution, foreign atoms replace some of the atoms in the original lattice. The positions that the foreign atoms take are not uniform, but at random throughout the lattice. It will be assumed that the lattice is a perfect one, that is, one without any vacant or unoccupied sites. Since the final equations of the quantitative theory can be shown to be usable for real crystals, this idealization is deemed valid.

As there are no vacant sites in the lattice parameter, it will be necessary to loosen two atoms before diffusion can occur. It is also necessary that one of the atoms be of the original species and one of the foreign species. If both atoms are adjacent atoms of the same species, self-diffusion occurs, and no resultant change takes place.

Two potential wells must be considered in this mechanism of substitution, one of which holds the original atom and the other which holds the solute atom. The depth and shape of the original well will change with the introduction of a foreign atom. Since the depth and shape of the well has changed, the original bond will also be changed. This new bond will be weaker

if the melting point of the solid solution is lower than that of the pure metal supplying the foreign atom. The new bond, however, can never be zero. Therefore, if C is the coordination number, the depth of the potential will wever diminish by more than 1/C in a dilute solid solution. When chromium atoms are substituted for iron atoms, the above conditions apply. Therefore, substitutional diffusion will be more rapid than self-diffusion.

Exchange, then, may occur when two adjacent atoms have acquired sufficient kinetic energy to become loosened. The time for the loosened atoms to exchange sites depends on the oscillations and the geometry of the lattice in the vicinity of the excited atoms. As a result, so many complications arise in calculating time from fundamental quantities that explanation will not be attempted herein. In the qualitative treatment, however, it is sufficient to point out that the time for exchange is independent of temperature and highly dependent on the geometry of the process.

As more foreign atoms are substituted for the original atoms, the potential wells of the original atoms are further reduced. Simultaneously, changes are taking place in the wells of the foreign atoms. When this occurs, Fick's law

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(dm = D.A.dt.dC) no longer applies because diffusion becomes a function of concentration.

It must be understood that the theory herein presented applies only to diffusion in an ideal lattice. Diffusion during the formation of a new phase was wholly omitted. It may be mentioned, however, that during the formation of a new phase, recrystallization will occur at its outer boundary with resulting columnar grains in the direction of diffusion because of this recrystallization. Upon the complete formation of the new phase, the laws of diffusion discussed prior to this section will apply to its individual grains. Furthermore, if the grain boundaries do not contain an agent that inhibits migration of atoms within them, diffusion along the boundaries will be greater than through the lattice, a fact due to the looser structure of the boundaries. Usually this type of diffusion is found in experimental observation.

Dushman-Langmuir Equation for Diffusion⁴

An equation applicable to diffusion in solids was formulated by Dushman and Langmuir in 1922. Their theory, based on the probability that an atom will jump from one atomic plane to a second, is exactly analogous to the probability of an unimolecular chemical reaction occurring. From the foregoing theory, the following equation was formulated.

 $-7-$

$$
D = K\delta^2 = \frac{E}{Nh}\delta^2 e^{-\frac{E}{RT}}
$$

Where D = diffusion constant,

K = probability that an atom will jump;

 s = the jump distance;

 E = the energy of activation per mole;

 $N =$ Avagrados number;

 $h =$ Plank's constant;

 e = the base of natural logarithms;

 $R =$ the gas constant; and

 $T =$ the absolute temperature;

A simplified form is obtained if

$$
A=\frac{\varepsilon \sigma^2}{N h}
$$

from which

$$
D=Ae^{-\frac{r}{RT}}
$$

This equation has been used in numerous experimental tests and has proved its validity and usefulness.

 \mathbf{r}

CHROMIZING OF STEELS

It is essential that the production of a layer of chromium on steels as protection against corrosion be applied on the whole surface, in a manner as uniform as possible. It is necessary to heat the object at diffusion temperatures of between 1000° and 1200°C (1830° and 2190°F). If the parts for chromizing are not finished parts, higher temperatures may be preferable. In the case of finished parts, the maximum temperature should not be above 1100°C (2110°F) if distortion and further work are to be avoided.

Since the rate of diffusion is constant, with suitable materials and a given temperature, the main difference in the different processes is the method in which the surface of the part to be treated is exposed to the chromium. Carriers in the solid, liquid, and gaseous state have been used either individually or in combination. Therefore, it is not only possible, but also important to select a good method from among the various processes; one that is favorable from the viewpoint of a corrosion-resisting coating, haximum chromium economy and availability to commercial practice.

Chromized surfaces that are resistant to

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scaling are not difficult to produce, but the production of chromium cases resistant to corrosive agents and acids depends on certain preliminary conditions. Inclusions or differences in concentration of chromium in small areas may cause weak spots. Good chromizing, therefore, makes desirable the avoidance of these weak spots, as the relatively thin coatings are easily corroded. These local weak spots can be avoided if the chromium layer is made thick enough, and if the chromium carriers are uniform throughout the surface of the part being treated. Moreover, in order to keep the formation of nonmetallic inclusions to a minimum, special care must be taken to select the carrier as well as the base material.

In the earlier-day process, chromium in the form of ferrochromium or granulated chromium metal, together with the piece to be treated, was heated to the diffusing temperature. This method did not produce a uniform chromizing effect. The method of using liquid metal baths⁵ has its limitations because of the high temperatures necessary for melting the chromium alloys. These temperatures are too high for chromizing finished pieces. Un satisfactory results are obtained with chromium metal in a gaseous state because of the still insufficient vapor pressure of the chromium metal. However, according to the equation

 $CrCl₂ + Fe \rightarrow FeCl₂ + Cr$,

which is the reaction of chromium salts with metallic iron, the gap between the two preceding methods can be filled satisfactorily. Because of the exchange reaction of chromium salts with iron, it is possible to produce a chromium metal surface on a piece of iron. Under these conditions the more favorable properties of chromium, namely, its relatively high vapor pressure, can be utilized to great advantage. When this method is used, it is essential to have a high concentration of chromous chloride and also a means of rapid removal of ferrous chloride formed in the reaction; otherwise, exchange comes to a stop. If the exchange reaction were allowed to come to equilibrium, it would be impossible to produce a pure chromium coating.

The only results obtainable in an equilibrium state would be a chromium concentration on the surface corresponding to the equilibrium position. The continuous migration of the iron towards the surface of the piece and the diffusion of chromium into the lattice of the piece being treated will insure against the stoppage of the chromizing at any certain chromium content. Thus, by keeping the equilibrium disturbed by the removal of ferrous chloride and the addition of chromium, the production of a chromium-rich case is possible. There are two other factors, beside the equilibrium position, which are dependent on temperature:

the removal of ferrous chloride and the rate of diffusion of chromium.

Chromous chloride salts can be produced either from the reaction of hydrogen chloride with ferrochromium or the reaction of barium chloride with pure chromium or ferrochromium. Chromous chloride has some very useful properties essential in the process of chromizing. The most important properties of CrCl₂ are that it fuses at 820°C (1508°F) and possesses a vapor pressure (abt. 25 mm.) at a temperature as low as 1000°C (2010°F). Chromous chloride vaporizes to a great extent at temperatures as low as 900°C (1650°F), which is an essential state for the diffusion of chromium into iron.

The main advantage of the salt bath process is that any desired concentration of chromium up to 50 per cent can be obtained in the liquid state. While in the gaseous state, chromium concentrations are quite limited because of the fact that in this condition the chromium content is a function of temperature and partial pressure. Therefore, the use of Cr or ferrochromium plus a chloride salt makes it possible to work with a specific optimum chromium supply, and hence thicker cases per unit of time may be obtained. Mr. Bennek, 1 et al., in their paper had this to report: "The most favorable conditions, which of course are

a function of temperature and the rate of diffusion, usually are obtained with a chromous chloride content of the salt bath of about 20 per cent." The writer, however, obtained very good results with a chromium content of 50 per cent by weight in a mixture of powdered-chromium metal and barium chloride.

In the foregoing discussion only one chemical reaction was assumed, that is, the one with chromous chloride and pure iron. There are, however, other chemical reactions that take place if the iron is an alloy of carbon, manganese, silicon, aluminum, or titanium. These alloying elements may have a favorable or unfavorable influence on the reaction.

In the case of low-carbon steels, the carbon diffuses into the high-chromium case at diffusion temperatures and accumulates as chromium carbides. This carbon diffusion impedes the diffusion of chromium into the steel, the result being a very thin case with a high-chromium content. Although the case has a high-chromium content, the chromium carbides are predominently those which lower the case's resistance to corrosion. Therefore, it is necessary to use either low-carbon steels (.01 per cent carbon) or to add some element such uss vanadium or silican to impede the diffusion of carbon into the chromium case. The addition of one of these elements makes possible the appreci-

able reduction of the concentration of carbon in the chromized case of steel containing up to 0.2 per cent carbon.

Although chromizing in the liquid phase has such special advantages as the attainable degree and the uniformity of concentration of chromium carriers, it does not have the potentialities of the gaseous state for producing a maximum uniformity of case.

DESCRIPTION OF EQUIPMENT

Diffusion was accomplished by heating the parts to be chromized in an electric tube-furnace. The quartz tube was 24 inches long, had an inside diameter of one inch, and was heated externally by chromel elements. To the loading end of the quartz tube was attached a twelve-inch, thin-walled, copper sleeve that served as a cooling chamber. Around the copper sleeve a tight coil of copper tubing was soldered so that water could be circulated around the cooling chamber to fascilitate rapid cooling. The following pieces of apparatus were then assembled into a chromizing train:

- 1. Hydrogen bottle in series with
	- a. pyrogallic acid washer
	- b. sulfuric acid washer
	- 2 U-tubes with calcium chloride c_{\bullet}
	- d. electric furnace
	- e. cooling chamber
- 2. Ammeter, millivolt meter, resistor, and thermocouple
- Standard metallographic laboratory and $3.$ dark room equipment

The hydrogen gas was passed through a pyrogallic acid washer to remove any oxygen that might have been present, through a sulfuric acid washer to remove the water, and through the

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calcium chloride U-tubes to insure the drying of the gas. The gas then passed into the electric furnace and cooling chamber. As the gas emerged, it was burned in order to safequard against explosions. This train made it possible to carry on chromizing and cooling in an hydrogen atmosphere. Illustration No. 1 shows the hydrogen train used in the study of chromizing carbon steels.

The millivolt meter attached to the thermocouple was calibrated against a standard Weston potentiometer and thermocouple.

The ammeter, resistor, and furnace were connected in series. It was thereby possible to regulate the temperature of the electric furnace at all times.

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PROCEDURE

Preliminary Investigation

In order to standardize the study of chromizing carbon steels and to make certain that satisfactory results might be obtained with this train, a preliminary run was made.

Since apparatus for the production of CrCl₂ was not available, it was decided to try using a mixture of BaCl_o and Cr; accordingly, an arbitrary mixture of 50 per cent BaCl₂ and 50 per cent chromium, by weight, was selected.

The bottom of a nickly boat was spread with the above mixture. Two steel samples were then placed in the boat, and more of the same mixture was packed upon and around the steel samples.

The boat, with an iron wire attached, was then introduced into the furnace. The wire was of such a length that it extended out through the cooling chamber at the end of the furnace. During the entire loading procedure, the hydrogen was flowing through the train. The samples remained in the furnace at 1780°F for one hour, after which time they were pulled back into the cooling chamber by means of the wire attached to the boat and allowed to cool in the hydrogen atmosphere.

The samples were then removed, mounted in lucite, polished, and etched in 5 per cent picral

for approximately thirty seconds for microscopic examination. The results obtained from the above procedure showed such definite possibilities that this method was adopted for further investigations.

Standard Procedure

The foregoing procedure was somewhat modified in the following manner:

100 grams of BaCl_o and 100 grams of Cr 1. were weighed out to approximately .01 of a gram.

2. The two constituents were mixed thoroughly with a rolling cloth. This mixture was then stored in an air-tight container.

3. The samples were packed in a nickle boat and chromized.

The chromized samples were mounted in $4.$ lucite and etched in 5 per cent picral.

5. Each sample was measured for depth of penetration.

6. Photomicrographs were taken of the armco iron samples.

Illustration No. 1 -- A view of the hydrogen train used in the chromizing investigations.

EXPERIMENTAL WORK AND RESULTS

Six different steels were used in this study; S. A.E. 1010, 1020, 1035, 1050, 1065, and armco. ingot iron. A sample of each steel was chromized for $\frac{1}{2}$, 1, 2, 3, 4, and 5 hours; therefore, a total of 36 samples were treated. These samples were chromized in a mixture of pure powdered chromium and barium chloride. Illustration 1 is a photograph of the chrom1z1ng apparatus used.

To remove the oxygen, tank hydrogen was passed through a washer containing pyrogallic acid; then, to remove water vapor, through another washer conta1ning sulfuric acid; and next, to make sure that the bydrogen was dry, through two U-tubes containing calcium chloride. The purified, dry hydrogen was passed through a fused-quartz tube furnace containing the specimens.

The procedure for each run was as follows. The furnace was heated to its maximum temperature, about 1780°F, after which the hydrogen was turned on and ignited at the exit of the train. A nickel boat containing six specimens packed in pure chromium and barium chloride was then introduced into the furnace.

When the samples had remained in the furnace the required length of time, the boat was pulled out of the furnace into the cooling chamber by

means of the wire attached to the nickel boat. The boat was allowed to remain in the cooling chamber until it was approximately roomtemperature. The specimens were then removed from the bath and mounted in lucite, polished, and etched with 5 per cent picral, which attacks only the ferrite grains. The diffused zones were measured to determine the depth of chromium penetration.

The results obtained from the study of the steels are given in Tables 1, 2, 3, 4, 5, and 6. After these results were examined. it was decided to make one more diffusion test in a furnace capable of attaining higher temperatures. The outcome of this operation is given in Table 7.

The temperature of the furnace referred to in Table 7 was 1850° F. It is apparent that with a rise in temperature of approximately 70° F the depth of the layer increased about 241 per cent. If each value for the depth of layer in Table 6 were multiplied by 2.41, a fair estimate of the depth of chromium at each of the tabulated times could be obtained for a temperature of 1850° F. From a close study of Tables 6 and 7. the examiner finds it possible to observe that a certain minimum temperature must be exceeded in order to accomplish proper chromizing. Above that critical minimum point, temperature may vary, but not to the extent that it critically influences

Illustrations 2, 3, and 4 are photomicrographs of the chromized layers and grains of ferrite. These illustrations are of the armco ingot iron specimen chromized at 1850° F. The columnar structure of the chromized layer was revealed with an etching solution of HNO₃, HCl, and $CuCl₂$

The chromizing results from Table 6 are shown in a time-penetration relationship in Figure $2.$

The specimens for the five-hour run were weighed before and after chromizing. The results obtained are given in Table 8.

Temperature - 1780° F.

TABLE 2

TABLE 4

TABLE 5

Temperature - 1850° F.

TABLE 7

Illustration 2 -- Shows a chromized layer produced on armco ingot iron after two hours at 1850° F. The ferrite was etched 5 per cent picral. Magnification, 200X.

grains magnified 200X and etched with 5 per cent picral. These grains show the structure of the chromium layer.

parts of HNO₃, 3 parts HC1, and 0.5 grams of CuCl₂. The etch solution corroded the ferrite grains beyond recognition.

Time-penetration relationship of chromium in armco ingot iron, diffusion temperature of 1780° F.

DISCUSSION OF RESULTS

The results obtained from the samples tested at 1780° F. were very inconsistent. There is no doubt that the inconsistency was due to the fluctuation of the chromizing temperature. This temperature, if maintained continuously, would have been sufficient. Later, however, it was discovered that this was impossible, because of the fact that other furnaces on the same power source were being used quite frequently during the chromizing runs. This fluctuation of current was especially noticeable in the five-hour chromizing cycle. Since time was of the essence, it was impossible to watch the furnace constantly.

It is doubtful that the carbon content had any influence on the results, for better results were obtained with the 1050 than with 1010 steel. According to all the theories, the diffusion of chromium becomes slower as the percentage of carbon in the steel is increased. The armco ingot iron was the only sample that fonformed to these theories.

That the temperature was too low was proved when two samples of armco ingot iron were run at 1850° F. The chromized layers obtained with this higher temperature wer 2.41 times as thick, or an improvement of 241% over those chromized

at 1750° F. If time had permitted, it is quite certain that the samples, if run at a higher temperature, would have shown a marked improvement.

Difficulty was experienced in polishing a sample containing two phases, such as chromium and ferrite. If the chromium were polished smooth, the ferrite grains would show signs of smearing, and if the ferrite were polished with care, the chromium would have streaks, as shown in Illustration 2.

Another problem was encountered in etching the samples. It was quite simple to etch the ferrite with 5 per cent picral; however, when an attempt was made to etch both chromium and ferrite, a quite different problem arose. After numerous attempts at showing the columnar structure of chromium and the structure of the ferrite grains. simultaneously, the decision was logically reached that they would be shown separately.

RECOMMENDATIONS FOR RESEARCH

Future investigations should be carried out in a furnace capable of attaining 1850° F. or above. Such a furnace would insure that a small fluctuation in current would not lower the temperature required for chromizing.

Since it is very difficult to obtain accurate measurements from a rough surface, the samples, before chromizing, should be polished, to give smooth edges free from burrs.

Interesting subjects for further study are suggested below.

1. The chromizing of alloy steels to find the effect of alloying elements on the diffusion rate of chromium into iron.

2. Tests to compare diffusion of chromium from a molten salt as compared with diffusion from a gaseous atmosphere.

3. X-ray studies of chromized layers to determine the extent of solid solubility of chromium in iron and also to determine the nature of the chromium carbides that may be present.

Tests to determine the corrosion re-4. sistance of a chromized surface as compared to the corrosion resistance of a chromium plated surface.

CONCLUSIONS

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From the results obtained, the following conclusions can be drawn:

The rate of diffusion of chromium into $1.$ iron is dependent upon the temperature; the higher the temperature the more rapid the diffusion.

The diffusion of chromium into iron is $2.$ accompanied by a slight increase in weight.

Chromizing involves a conservation of $3.$ the valuable chromium metal, for only a thin layer of the chromium-rich alloy is needed at the surface to give corrosion resistance.

4. Plain carbon or inexpensive alloy steels, if chromized for protection against corrosion, may be used in place of the more expensive stainless steels containing 18 per cent chromium and 8 per cent nickel.

The chromium-rich layer at the surface, $5.$ if produced by diffusion of chromium into iron, is an integral part of the base metal and therefore shows no tendency to chip or peel as does an electrolytic deposit of chromium.

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