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264612
61-4-6

INVESTIGATION OF THE HEAT CORROSION RESISTANCE AND
STRUCTURE OF A NUMBER OF IRON-BASE ALLOYS, RELATIVE
TO THEIR COMPOSITIONS

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English Pages: 12

SOURCE: Issledovaniya Po Zharoprochnym Splavam, Moskva,
Vol. 6, 1960, pp. 259-267.

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CORROSION/
INVESTIGATION OF THE HEAT RESISTANCE AND STRUCTURE OF A NUMBER OF IRON-BASE
ALLOYS, RELATIVE TO THEIR COMPOSITIONS

by

S.D.Gertsriken, I.Ya.Dekhtyar, L.M.Kumok

The heat-resistant materials of today consist of alloys based on a transition group in Mendeleev's periodic system. The introduction of various alloying elements, even in small quantities, has a significant effect upon the heat resistance of steel. Therefore, the heat-resistant alloys employed are to a considerable degree exceedingly complex in chemical compositions. Usually XX phase equilibrium is achieved only after a very long period of holding in this type of alloy even at high temperatures.

Table 1

a) Alloy number; b) Content of elements, wt.%

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This circumstance is due to the extreme slowness of diffusion processes. At operating temperatures, the properties of these alloys change constantly as a consequence of variation in their phase composition and the structural components of the individual components. Therefore, before testing the mechanical properties of modern alloys designed

for service under high temperatures, it is necessary to study the phase composition and kinetics of such alloys under various conditions. ~~THESE~~

The present paper is devoted to a search for new heat-resistant alloys based on iron and suited to long-term service at elevated temperatures as gas-turbine parts.

Alloys (ingots of 0.3 - 0.5 kg) were prepared ~~XX~~ in an argon atmosphere in a high-frequency vacuum furnace, ^{from} pure metals, whose composition is presented in Table 1, having been used.

The alloys melted were subjected to an ^{homogenizing} ~~homogenizing~~ anneal at 1200° for 50 hrs, and were quenched in water from that temperature. They were then forged and rolled to 5 - 6 mm in the 950 - 1050° temperature interval.

Investigations of the microstructure of the quenched specimens showed that alloys Nos. 1 - 5 containing 5% Al constituted a two-phase solid solution of the ferritic - austenitic type. The phase ratio in these alloys varied, and alloys Nos. 6 - 19 constituted ~~X~~ single-phase solid alloys of the ferritic type.

Corrosion) A Study of Heat Resistance and Weldability of Alloys

Investigation of the heat resistance of the alloys made use of flat specimens with ground surfaces. The specimens ^{were} first weighed and suspended ^{vertically} from a ~~XXXXX~~ nichrome wire in a ¹/₂ hrs furnace and, after heating, were cooled along with the furnace. They were then weighed on microanalytical ^{balances} scales with an accuracy of $\pm 1.25 \times 10^{-5}$ gm. The degree of heat resistance was determined from weight gain.

The investigations were run at a temperature of 1000°. The results obtained for the index of corrosion resistance are presented in Fig. 1. As we see, the heat resistance of two ferrite-austenite alloys Nos. 1 - 4 is considerably less than that of the single-phase ferritic alloys Nos. 6 - 11. At the outset oxidation goes somewhat more rapidly, but thereafter its speed remains virtually unchanged. Both in the single-phase ferritic

region and in ~~the~~ the two-phase austenitic-ferritic, the presence of Zr in the alloy greatly diminishes heat resistance. Moreover, the greater the amount of Zr the lower

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$$\frac{gm}{m^2 \cdot hr}$$

$$\frac{gm}{m^2 \cdot hr}$$

hrs

Fig.1 - Change in Weight Gain Depending upon the Time ^{Heating}
for which ^{the} ~~the~~ Alloys were Held.

1 - No.1; 2 - No.3; 3 - No.4; 4 - No.7; 5 - No.8; 6 - No.11

261 the heat resistance. The presence of Nb reduces heat resistance to an insignificant degree. Alloys Nos.6, 7, and 11 have the best heat resistance.

Weldability tests were performed on alloys Nos.12 - 19, in the form of forged and rolled slabs 2 - 5 mm thick, 25 - 40 mm wide, and 70 to 100 - 120 mm long*. The weldability tests provided for fusing beads to the surfaces of the plates and making ~~one- and two- tee-joint fillet welds~~ one- and two- tee-joint fillet welds (the tees were made only of plates of 4 - 5 mm thickness). The fusing of the beads and welding of the tees were performed by inert gas shielded ^{gas} welding using a nonconsumable electrode without filler material. The electrode was tungsten, 1.7 - 2 mm in diameter, and the gas was pure argon.

As a result of the preliminary weldability tests it may be held that alloys Nos.12, 13, and 14, containing approximately 2% Si, 3% Mn, and 3% Al weld well and better than any of the other test alloys. Further investigations were performed with alloys

*The weldability tests were performed by B.I. Medovar, senior research assistant at the Ye.O. Paton Electric Welding Institute of the Ukrainian Academy of Sciences.

Nos. 12, 13, and 14, whose resistance was studied at temperatures of 800, 900, and 1000°. Moreover, an investigation was made of the heat resistance of alloy No. 12 at 1200° for 100 hrs and of alloy EI435. The results obtained (Fig. 2a, b, c) show that the rate of oxidation of alloy No. 12, which is 1.01 gm/m²/hr. is significantly less than the rate of oxidation of alloy EI435, which is 1.87 gm/m²/hr.

At high temperatures, a thin, strong and dense film of oxide forms on the surface of the specimen. It, ~~like~~ like the metal, undergoes oxidation, and constitutes a solid solution of oxides of aluminum, iron, and chromium. The fact that heat resistance is lower at 900° than at 1000° may be explained by the fact that at 900°, the alloys studied consist of a mixture of two phases, α and γ , whereas at 1000° they consist of a single-phase γ -solid solution.

If the data on the heat resistance of these alloys are compared with that of other known iron-base alloys employed as heat-resistant material, EI435 for example, their heat resistance proves to be higher: the weight gain of EI435 at 1000° in 100 hrs is 0.4 gm/m²/hr, whereas the gain in the alloys under investigation is 0.2 gm/m²/hr (Bibl. 1).

Moreover, ferrite alloys Nos. 633 and 704, whose composition is presented in Table 2 were studied for heat resistance and heat corrosion resistance.

Table 2

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a) Alloy number; b) Content of elements in wt. %

The results of the investigations for heat corrosion resistance performed at 1000, 1100, and 1200°, are presented in Table 3.

hrs

$$\frac{\text{gms}}{\text{m}^2 \text{ hr}}$$

hrs

$$\frac{\text{gm}}{\text{m}^2 \text{ hr}}$$

hrs

Fig.2 - Change in Weight Gain Relative to Holding Time

~~XXXXXX~~ at 1000 (a), 900 (b), and 800° (c)

of the Following Alloys:

1 - No.12; 2 - No.13; 3 - No.14

a) Alloy number; b) Temperature, °C; c) Area, cm²; d) Duration of test, hrs

As we see, oxidation goes somewhat more rapidly at the outset, but then its speed becomes approximately identical throughout.

As a result of oxidation, a dense film of silvery oxides appears on the sample. It is obvious that these are ~~essentially~~ chiefly aluminum and chromium oxides. If we compare these alloys to other iron-base alloys it will be found that their heat corrosion resistance is higher. Thus, for example, (Bibl.1) shows the weight gain of chromium-nickel-molybdenum heat resistant steel Kh16N26G1 and 5S1M6 to be 0.2 gm/m²/hr at 200°, whereas with the alloys under consideration it is 0.06 gm/m²/hr at 1100°. At 1200° alloy No.633 performs better than alloy No.704 and better than a heat corrosion resistant alloy of approximately the same chromium and aluminum content, described in (Bibl.2).

Stress-rupture strength was investigated for alloys Nos.633 and 704. The tests were run on a tensile testing machine. ~~These specimens,~~ ~~These specimens,~~ having first been subjected to heat treatment, were gripped in the clamps used for that purpose, the bottom door of the furnace was closed, and the furnace was slowly brought to a desired temperature. Then, after holding for 10 min, the specimens were placed under load ~~and~~ and tested to rupture.

The specimens studied had been quenched from 1200° and aged at 100° for 2, 4,

and 6 hrs. Alloy No. 704 was tested at 700, 760, and 800°, and under a load of 11.6 kg/mm². As quenching temperature was increased, the stress-rupture strength of the alloy rose considerably. With increase in aging time, the stress-rupture strength rises, achieving a maximum at 4 hrs of aging (at 800°), whereas it drops if aging is continued further.

When test temperature is reduced from 800 to 760°, the stress-rupture characteristics improve to an insignificant degree, but diminish sharply with further reduction in service temperature. It may be stated that 700° is the optimum service temperature for this alloy.

When No. 633 alloy was tested, specimens quenched from 1200° and aged at 800° for various periods of time were employed. A rational ~~heat~~ heat treatment for this alloy is quenching from 1200° with holding for 10 hrs and aging at 800° for 9 - 10 hrs.

We have determined the relationship between the stress-rupture characteristics of No. 633 alloy ^{and the} ~~to~~ temperature ^{at} ~~in a~~ constant ~~XX~~ stress of $\sigma = 12 \text{ kg/mm}^2$, and ~~XX~~ relative to ~~XXXX~~ stress applied at a constant temperature of 760°. Above a test temperature of 750 - 760°, the strength of the alloy drops sharply, exponentially. ^{the} Heat resistance is highly dependent upon the stress applied. If at $\sigma = 15 \text{ kg/mm}^2$, the stress-rupture characteristic is 25 hrs, an increase in the load to $\sigma = 19 \text{ kg/mm}^2$ diminished it to 4 hrs 5 min.

264 A comparison of the results with the experimental ferritic alloy No. 633 and an austenitic ~~XX~~ of type 14-4, containing 0.22% carbon quenched from 1200° shows that at a service temperature of 700° and a load of $\sigma = 10 \text{ kg/mm}^2$, the stress-rupture characteristic of an austenitic alloy is approximately 250 hrs, while that of the ferritic alloy at the same temperature and load of $\sigma = 12 \text{ kg/mm}^2$ is ~~XXXXXX~~ ^($\sigma = 12 \text{ kg/mm}^2$) is ~3600 hrs.

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X-Ray Structural Studies

X-ray structural analysis was performed in chromium radiation and RKD-17 Dbye cameras. The mean parameters of ~~the~~ lattice after various aging periods at 800°C are presented in Tables 4 - 6.

Table 4

- a) Aging time, hrs; b) Lattice parameter, KX; c) Alloy No.1; d) Alloy No.2;
e) Quenched

As is evident from Table 4, the lattice parameter of the alloys diminishes as aging time increases, increases somewhat in the 6 - 30 hr range, and ^{then} again diminishes (in the case of alloy No.1). The reduction in the lattice parameter obviously relates to the separation of excess phases, and ^{an} increase ~~in~~ therein relates to ^{their} dissolution ~~thereof~~. Dissolution is confirmed by study of the microstructure. An uneven etchability of various zones of the grain, i.e., a process whereby the grain becomes nonuniform in composition in the aging process, is observed. The x-ray data also testified to the fact that when aging occurs at constant temperature, ^a continuous exchange of elements between the solid solution and the secondary phases occurs.

Many supplementary lines may be seen in the radiographs of alloys Nos.1 and 2. After a special analysis of the separated phases was performed, it was found that, in the case of alloy No.1, these lines belong to the separated carbide phase ~~XXXXX~~ Cr₂₃C₆, whereas for alloy No.2, both of them ~~XXXXXX~~ belong to the σ-phase (as is confirmed by X comparison ^{with} a radiograph obtained from the pure phase), while the remainder

belongs to some new phase. The lines characteristic of the new phase also exist on the radiographs of alloy No.2. They appear only after aging, ^{and} are not to be seen in the ~~MM~~ quenched condition. After analysis it was found that these lines may be ascribed to interference from the (411) plane, and that the computed parameter is $4.36 \text{ \AA}$, which is in approximate agreement with the chemical compound FeSi.

Lines of a body-centered lattice are present in the radiographs of hardened alloys. Even ~~within~~ ³⁰ 27 hrs of tempering, the radiographs also show lines of a face-centered γ -lattice, which becomes ^{more intense} ~~ever brighter~~ as further tempering time passes.

As is evident from Tables 5 and 6, the lattice parameters ~~XXXXX~~ of all the alloys pass through minima at various times. If one follow the course of variation

Table 5

Variations in the Lattice Parameter of the Principal Phase

- a) Aging time, hrs; b) Lattice parameter, KX; c) Alloy No.4; d) Alloy No.5; ~~MM~~
e) Alloy No.6; f) Alloy No.7; g) Alloy No.8; h) Quenched

Table 6

Variation in the Lattice Parameter of the Separated ~~XXXX~~ Phase

- a) Aging time, hrs; b) Lattice parameter, KX; c) Alloy No.4; d) Alloy No.5; e) Alloy No.8

of the parameters of the major and the separated phases, it will be evident that the course of the variation of the parameters with aging time is contradictory, to wit: reduction in the parameters of the ferrite lattice corresponds to an increase in the parameter for the austenitic phase, and vice versa. This fact is apparently capable of being explained by the fact that the transfer of atoms of large radius from the ferritic to the austenitic phase occurs with the decomposition of the basic solid solution. In view of the fact that the smallest parameter is quite large, aluminum, niobium and zirconium, whose atomic radii are 1.43, 1.47, $\text{\AA}$ and 1.6 $\text{\AA}$ respectively, whereas the atomic radius of the remaining components is 1.25 $\text{\AA}$, may very well be the elements here concerned. Moreover, the bulk of the change may apparently be ascribed to the aluminum transition.

Tables 7 and 8 present the mean values of the lattice parameters of the initial and the separated phases of alloys Nos. 9 - 11 after various aging times, whereas Table 9 presents the values of the parameters of the initial phase of alloys No. 12, 13.

The radiographs of tempered alloys Nos. 9 - 11 indicate the presence of austenite and ferrite lines. In the process of aging at 800°C, the ferrite lines disappear, a σ -phase separates out, as do certain other phases. The lattice parameter of the initial solid solution of austenite drops sharply to 100 hrs, and subsequent thereto it remains constant for alloys 9 - 11. This testifies to the stability of the phase composition.

The lines of a face-centered lattice are present in the radiographs of cast alloys Nos. 12 - 13. As tempering proceeds, the lattice parameter of the base varies, this variation differing for various alloys. In the case of alloy No. 12 which contains much carbon, it gradually diminishes. This testifies to the fact that the solid solution is impoverished in alloying carbide-forming elements. In alloy No. 13 ("carbon-free")

Table 7

Variation in Lattice Parameter ~~XXXXXX~~ KX of Principal Phase

a) Alloy number; b) Quenched; c) Aging, hrs

Table 8

Variation in Lattice Parameter ~~XX~~ KX of Separated Phase

a) Alloy number; b) Quenched; c) Aging, hrs

Table 9

a) Alloy number; b) Cast; c) Aging, hrs

some increase in lattice parameter occurs. One might ~~XXXXX~~ assume that, in this alloy, reduction in the ~~XXXXX~~ lattice parameter proceeds in two opposing directions: the first direction, related to the decomposition of the solid solution causes a reduction in the parameter. The second direction, relating to the appearance of internal stresses is due to the suppression of the dispersed nuclei of an excess phase having a greater specific volume than the principal solid solution.

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These supplementary lines obtained on the radiograms of alloys Nos. 12 and 13 belong to carbides of chromium and the σ -phase. It is significant that the σ -phase appears in various alloys under nonidentical tempering conditions, that carbon accelerates the decomposition process, and that the lines for the σ -phase appear in alloy 12 considerably sooner (2 hrs of aging ^{instead of 12 hrs} ~~INSTEAD OF 12 HRS~~) than in the case of the carbon-free alloy.

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