Research and development work on substitute electrical resistance alloys for heating elements

R. CHOUBEY, S. CHOUDHURI, B. N. DAS and B. R. NIJHAWAN

FROM the start of the Second Five-Year Plan great emphasis has been laid on production and utilisation of electric power in various industrial and domestic appliances. Electric heating is thus gradually replacing solid-fuels, gas and oil heating. Increasing application of electric heat with all its attendant advantages will fail to register full impact unless suitable electrical heating elements, having long high temperature service life are indigenously available at reasonable cost.

Conventional types of heating elements[1,2,3] used for domestic or industrial heating applications contain high percentage of nickel and some cobalt. Resources of nickel and cobalt hardly exist in India. Thus the entire requirement of heating elements used in India is imported, involving heavy drainage of foreign exchange.

With a view to developing electrical heating elements free from non-indigenous nickel and cobalt, comprehensive research and development work have been under way at the National Metallurgical Laboratory for several years resulting in the development of electrical heating elements based on mainly indigenous raw materials.

Desirable characteristics that are needed for the finished product of electrical heating elements are the following :

1. High electrical resistivity and low temperature coefficient of resistivity.
2. Good reaistance to scaling and oxidation above the red hot temperature.
3. Low coefficient of thermal expansion.
4. Comparatively high melting point and low meltting range.
5. High temperature strength and stability of structure at elevated temperature.
6. Low specific gravity, and
7. Good cold drawability of the material to enable final cold drawing operation to produce fine gauges of wire.

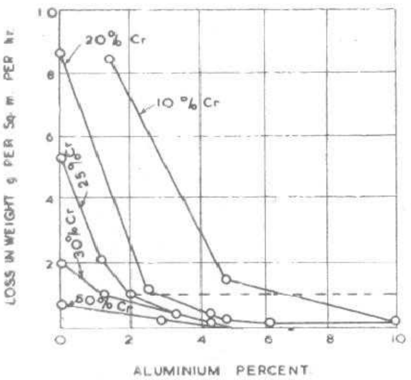
Messrs R. Choubey, S. Choudhuri, B. N. Das, Scientists, and Dr B. R. Nijhawan, Director, National Metallurgical Laboratory, Jamshedpur.

It has been known that[4,5] Cr and Al when alloyed with iron enhance the electrical resistance and resistance to scaling and oxidation at high temperature. Aluminium (Fig. 1) has been found to exert a more pronounced effect than Cr. These considerations have led to the adoption of Fe-Cr-Al system of heating element.

Reference may be made to the ternary Fe-Cr-Al system (Fig. 2). Both Cr and Al have considerable solid solubility in iron. They both form with Fe closed γ loop type binary system leaving single α phase up to the melting point in the workable and useful composition range. The scaling resistance of these alloys has been explained on the basis of formation of thin, continuous, adherent and refractory scale on the surface. The chief difficulty with this type of alloys is their strong tendency towards grain growth during use at elevated temperatures rendering the metal thereby weak and brittle. So at each stage of hot working, extreme care has to be exercised in controlling the tempereture and amount of working closely following predetermined mechanical working and heat-treatment cycles. Furthermore, extremely deletarious effect of carbon, other impurities and inclusions on scaling resistance and workability of these special alloys necessitate their avoidance through proper control by use of selected base melting charge and taking recourse to carefully controlled melting and casting techniques. Pouring temperature exercises considerable influence on the cast structure and hot-workability of the alloy ingot.

Use of certain elements[6] in small quantities within certain limits has been found to considerably improve workability without deletarious effect on scaling resistance and also help to inhibit grain growth during high temperature service of the heating elements.

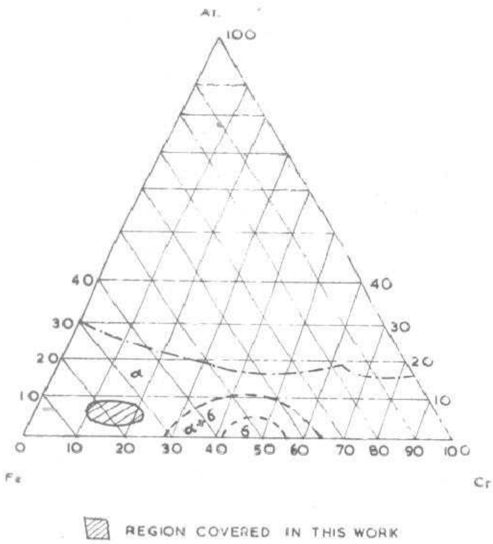
Successful development of Fe-Cr-Al alloys of optimum composition has been based essentially on Indian alloy-



1 *Effect of Cr and Al on oxidation resistance of Fe-Cr-Al alloys (From kornilov[4])*

ing elements with suitable additions of those minor alloying elements in optimum combination to obtain maximum ease in fabrication and final requisite high temperature properties and service life. Detailed study has also been made to formulate optimum melting and casting techniques, and hot and cold workability of the alloys. Depending on the maximum service temperature, minimum percentages of Cr and Al content are required in optimum proportion to satisfy the requirements of electrical and scaling resistance. The selection of charge was also found to be critical. It has been already mentioned that carbon has a major influence in adversely affecting the scaling resistance of the Fe-Cr-Al alloys, and hence must be limited to a low level.

The alloying elements used besides Cr and Al were Mn, Si, Zr, Ti and misch metal in various proportions. Zr and Ti were chiefly intended for refining the grain size and restricting the grain growth at high temperatures. Some new elements like Cu, B, N, P, etc. were tried but the workability was found to be impaired by their additions.



2 *Condensed ternary diagram of the Fe-Cr-Al system (From kornilov[4])*

Initial phase of work was directed in the range of high chromium and low aluminium (alloy 4E6, 2E28) but was ultimately diverted to the range of low chromium and high aluminium with other minute additions (viz. alloy E31, 4E31).

It has been seen that manganese in small amount up to 2% has been found not to impair scaling resistance of these alloys to an appreciable extent but, on the other hand, is advantageous from the point of view of improving working properties. Silicon which increases electrical resistance to a great extent also causes excessive grain growth and embrittlement was not used in large amount. The effect of Zr, Ti and misch metal (rare earth) additions in small quantities was found to show improved workability and good scaling resistance.

Experimental procedure

High frequency furnace was used for making the experimental heats. A large number of heats were prepared using low residue iron (C$\leqslant$·04%), low carbon ferro-chrome, ferro-silicon, electrolytic manganese, commercially pure aluminium, ferro-zirconium, ferro-titanium, misch metal, etc. as raw materials. In a few cases an Fe-Cr-Al master alloy prepared earlier for the purpose with suitable Cr-Al ratio was charged with a view to minimising alumina content in the heat which have been found to affect workability adversely. The presence of large amount of alumina inclusions was observed to be one of the reasons for unsatisfactory ductility as well as cracking during cold drawing operation. The range of composition covered in the work is given in Table I.

TABLE I Range of chemical composition (%) of the experimental alloys

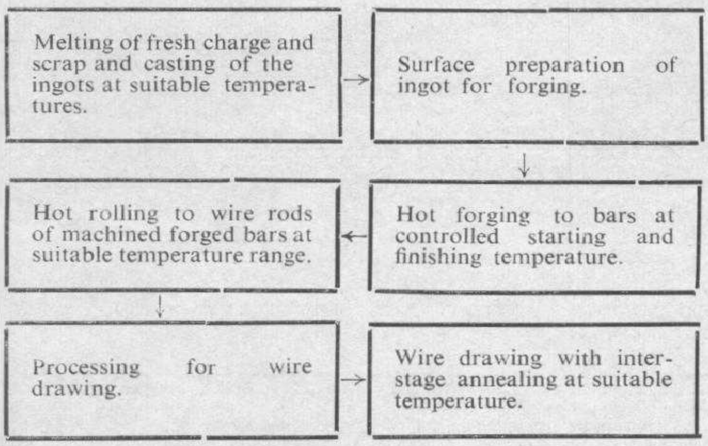
Cr	Al	Zr	Ti	Misch metal	C	Mn	Si
10–20	3–8	0·1–·3	0–0·3	0–0·3	0·04 max.	0·3 max.	0·6 max.

In most of the cases, ten kilograms heats were made which were cast into 2″ sq. ingots. The molten metal was either poured through a ladle or, directly poured into the ingot mould using hot-top practice. Some of the cast ingots, after stripping from the ingots moulds, were slowly cooled in a furnace kept at a temperature of about 1000°C. The hot-top portion including the pipe of the ingot was cut off. The ingots were then machined to remove the surface defects.

The ingot was then heated gradually to 1150°C and soaked for sufficient time. It was then hot-forged to about 1″ sq. bar. Finishing temperature was kept low to avoid coarse grain structure. The bar was again shaped and ground to remove surface defects if any, and finally hot-rolled to 3/8″ dia. rod in a bar rolling mill. Due care was taken to closely control the finish rolling temperature as the latter had pronounced effect on the final grain structure which, if coarse, greatly affected the drawability of the material. The rods were then pickled and finally the surface defects were removed by grinding. Cold wire drawing was carried out through successive dies down to the required gauges. It was found out that interstage annealing was necessary after 2/3 passes i.e., after about 30/40 per cent cold reduction. Suitable lubricant had to be developed before the wire could be drawn successfully.

All the stages of production starting from selection of charge to the finished wire as covered in this investigation is depicted in the flow sheet given in Fig. 3.

Figure 3 Flow sheet



Results and discussion

The difficulties in production of electrical resistance alloys with lower chromium and higher aluminium content than those in the conventional kanthal group of resistance alloys are known to be mainly in the poor workability of such alloys. In order to find out the factors which affect workability as well as the conditions which improve it, careful experiments were carried out at all stages of production varying the melting and casting techniques, hot working conditions and effects of various minor alloying additions on workability and final micro-structure and resulting physical and mechanical properties of the experimental alloys. Preliminary experiments revealed that grain size, non-metallic inclusions and hot finishing temperature profoundly affected the quality of the product. A thorough study was therefore made of macro and micro-structure at various stages of production.

Metallographic examination was carried out mainly for determination of grain size, non-metallic inclusions and other internal defects in the structure especially with a view to ascertaining and minimizing the cause of longitudinal cracking during wire drawing. The micro-structure consisted of single α phase structure. Some non-metallic inclusions (Al_2O_3) were found to be dispersed throughout the matrix. Grain boundary precipitation of some phase was seen in some cases. Considerable grain refinement was observed in cases where the working or heat treatment was done at a relatively lower temperature.

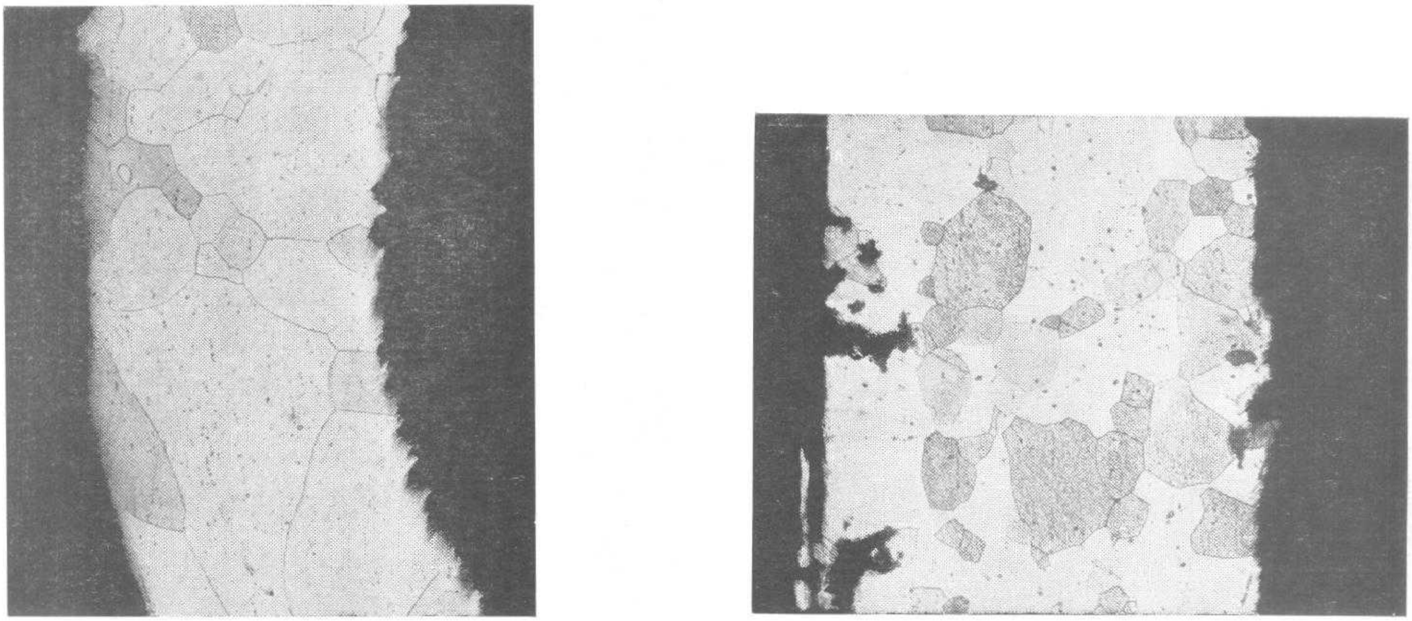
It was found that the ingots showing very coarse as-cast structure were susceptible to cracking during working. In some cases, longitudinal cracking was also noticed during drawing. The cracks penetrated deep to the centre of the wire. It appeared that these cracks were present in the rod during hot working stage. Careful romoval of these cracks at the stage of forged bar by machining or grinding was helpful to minimise the trouble. Some of the hot-rolled bars behaved in a brittle manner. Slow cooling and reheating the ingots to avoid thermal shock and precautions during melting and subsequently mechanical working and interstage annealing were found to be helpful to remove these defects. The metallographic examination of these defects showed that it was prominent in the bars with considerable inclusions.

A comparative evaluation of the ductility of these alloys as affected by grain growth resulting from soaking at high temperatures was made by simple reverse bend tests. The wire specimens were soaked at 1000°C for 15 hours. The results of the test are given in Table II where it would be seen that, for the temperature and soaking periods employed, the new alloys behaved almost in the same way as conventional heating elements. However, further tests for prolonged period of heating are yet to be carried out.

TABLE II Bend ductility under various heat treatment

Material wire 28SWG	Number of 180° reverse bends on 2·5 mm radius under conditions of following treatments: Cold drawn	Annealed (750°C) ½ hours	Soaked at 1000°C for 15 hours
E31	11	16	11
4E31	15	18	12
4E6	7	14	8
2E28	10	16	10
Kanthal DS	—	16	8
Nichrome	—	15	8

Experiments have been conducted to ascertain the grain growth tendency of these alloys on prolonged heating at higher temperatures. The samples from heat Nos. E31 and 4E31 were subjected to soaking at 1200°C for different periods extending up to 192 hours and their micro-structures studied at various stages. It was noticed from Fig. 4 that grain growth in E31 which



Alloy 4E31 × 100 *Alloy E 31* × 100

4 *Variation of grain size of alloy E31 and 4E31 after 192 hours at 1200°C*

has lesser content of aluminium was comparatively less.

The mechanical and physical properties including the accelerated life test of the wires were determined as given below in Tables III to V together with the corresponding properties of conventional heating elements wherever available. All the heats gave fairly good elongation per cent in tensile testing. From the results of tensile tests, it was found that the strengthening effect of aluminium was more than that of chromium. These tests also revealed that relatively low temperature forging resulted into considerably higher elongation and reduction of area values compared to those forged at higher temperatures. Consequently low finishing temperature resulted in a product more easily drawable.

TABLE III Tensile and hardness test results of experimental alloys

Heat No.	U.T.S. Tons/ sq. in	Elongation% (GL= $4\sqrt{A}$)	Reduction in area %	Hardness HV_{30} As cast	Hardness HV_{30} as hot forged
E31 (lower Cr)	59	16	55	—	191
4E31 ,,	50	20	62	207	219
4E6 (Higher Cr)	48	32	67	195	246
2E28 ,,	58	20	60	203	214

TABLE IV Electrical resistivity at various temperatures

Material	Electrical Resistivity (michrohm-cm) at temp.°C: Room Temp.	200	400	600	800	1000
E31	136·3	138·1	141·6	147·8	152·1	153·9
4E6	140·2	141·9	144·2	148·1	149·8	151·5
2E28	146·2	146·7	148·6	151·7	153·0	153·8
Kanthal	131·1	133·0	136·0	141·1	143·4	144·9
Nichrome	128·4	131·4	133·3	135·3	136·2	—

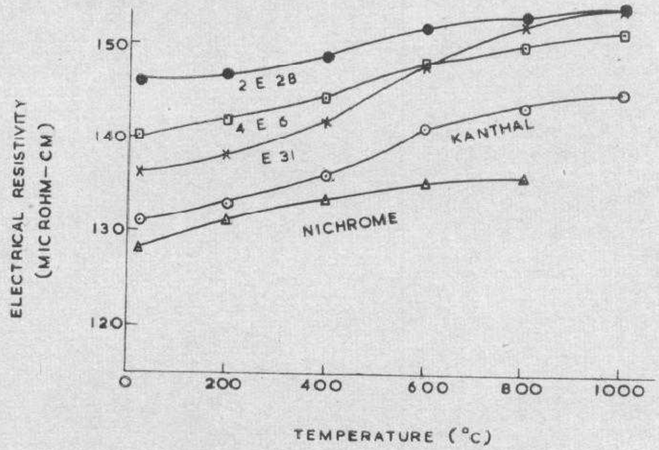
The electrical resistivity of wires has been measured at room temperature and elevated temperatures and reported in Table IV together with those of conventional heating elements. It was noticed from the resistivity temperature curve (Fig. 5) that the rate of increase of resistivity in the range of 400°C–600°C is rather higher, but the curves flattened out at higher temperature which is a desirable feature of such alloys.

Accelerated life test [8,7] of the heating elements was conducted as per A.S.T.M. Standard under 2 min. on and 2 min. off conditions. In this method (apparatus shown in Fig. 6) the test specimens of about 12″ long and 22 SWG dia. were heated by passing electric current through an automatically controlled voltage supply

TABLE V Accelerated life test result

Material	Accelerated life test results (hours) to burn out at different temperatures	
	1150°C	1200°C
E31 (Lower Cr higher Al series)	166	90
4E31 ,,	260	98
4E6 Higher Cr lower Al series)	70	42
2E28 ,,	90	60
Kanthal DS	310*	165*
Nichrome	145	67

* From Kanthal Hand Book (1954)

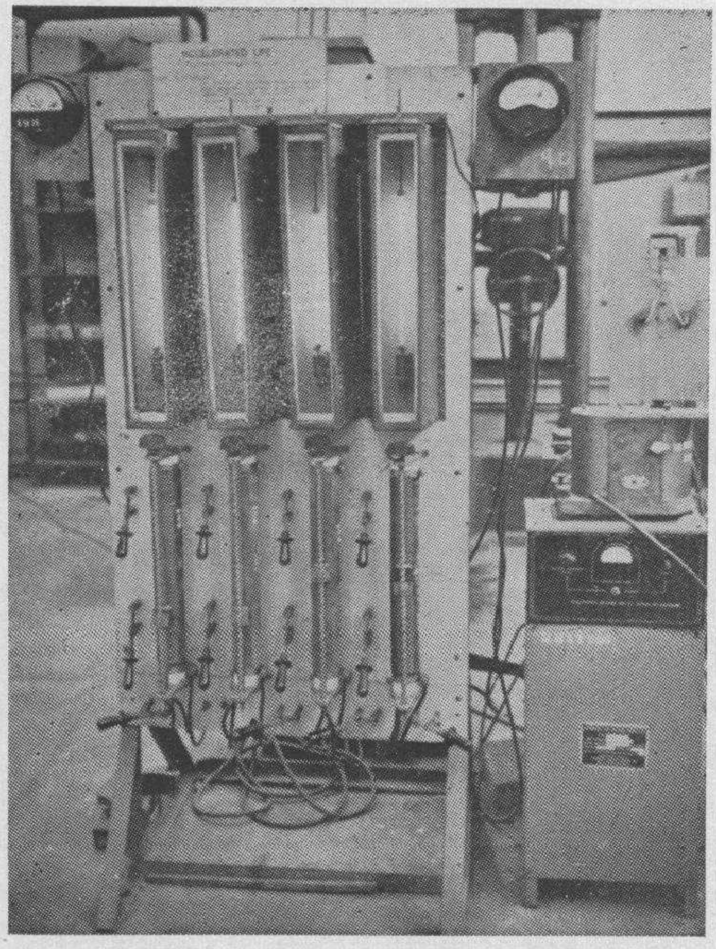


5 *Variation of electrical resistivity with temperature*

source. Temperature was measured by optical pyrometer. The results of the accelerated life test of the various experimental wires have been reported in Table V together with stmilar test results fo nichrome and kanthal DS. The life of some of the earlier heats was extremely low. Investigation to the cause for unusually low life of the particular heats revealed that large amount of inclusion, low aluminium content and high carbon and manganese were detrimental to the quality showing the necessity of avoiding these in the alloys.

The most important test for the quality of electrical heating elements is the life test which directly gives the service behaviour of the elements. When all the factors which adversely affect the workability and the properties of the substitute alloy investigated were reduced or eliminated as detailed above the property of the resulting alloy (E31 series) were comparable to the conventional heating elements though the total quantity of alloying elements in the substitute alloy was much less than that in the conventional alloys.

Though the quality of the alloy was found to be satisfactory, commercial utilization would depend on the cost of production. Total quantity of costly alloying elements in the alloy being comparatively small it could be guessed that the production cost would be much less than that of conventional heating elements. The cost of production of finished electrical resistance alloy of a particular gauge of wire was calculated out on the basis of a modest production of about 60 tons per annum, and taking into account the cost of raw material, utility, factory over-heads, depreciation, indirect cost and wages, etc. This worked out to be about Rs 12 per lb which is far less than cost of imported heating elements. So, in all respects the substitute alloy developed is expected to have good future.



6 *Accelerated life testing apparatus*

References

1. G. Nordstroem, Alloys good at temperatures above 2100°F. Metal Progress, Vol. 28, Oct. 1935, pp. 68–69.

2. Fred, P. Peters, Ni-Cr Alloys for Electric Resistors, Metal Progress, Oct. 1935, pp. 63-67.
3. R. J. Fabian, How to Select Resistance Heating Alloys, Materials in Design Engineering, Vol. 47, No. 2, Feb. 1958, p. 104.
4. I. I. Kornilov, Alloys of Iron-Chromium-Aluminium Poligrafhniga, Moscow, 1945, 192 p. (in Russian).
5. S. L. Case, Russians have new Heat Resistant Alloys, Iron Age, Vol. 167, March 22, 1951, p. 65-69.
6. K. Shinzi, Some Experiments on Fe-Cr-Al Electrical Resistance Alloys, Journal of the Japanese Instt. of Metals, June, 1949, 13, pp. 47-50, (in Japanese).
7. ASTM Standards, 1961, Life test, Accelerated, of Fe-Cr-Al-Alloys for Electrical Heating, B78,-59T.
8. N. V. Semenova and L. L. Shukov, Assessing the quality of alloys for electric heaters, stal, July, 1959.

Discussions

Mr **L. J. Balasundaram** (NML) :

(i) Was any superlattice structure observed in these alloys ?

(ii) Were the alloys aged, if so was there any change in electrical resistance with time ?

Authors :

(i) Superlattice structure was not examined in these alloys.

(ii) We did not study the effect of aging time on the electrical resistivity of the alloy directly. However, while carrying out experiment on acclerated life testing of these alloys it was observed that long time heating of the alloy at the service temperature resulted in decrease of heating current which had to be adjusted to a higher level in order to get same temperature of the samples. This indicated that there was an increase in the overall electrical resistance of the sample—part of which was attributed to change in the dimension of the wire and part to the increased resistivity resulting from prolonged heating.