

F84-10

MICROSTRUCTURAL CHARACTERISTICS OF CABOT ALLOY NO. 214,

A NEW SUPERALLOY DEVELOPED FOR APPLICATIONS IN

HOSTILE, HIGH-TEMPERATURE ENVIRONMENTS

by

K. V. RAO

R. B. HERCHENROEDER

The Metallurgical Society and American Institute of Mining, Metallurgical, and Petroleum Engineers, Inc. are not responsible for statements or opinions in this publication.

Papers delivered before meetings of The Metallurgical Society of AIME become the property of the Society, and are not to be published without written Society permission. Paraphrasing is permitted in editorial review, provided credit is extended to The Metallurgical Society of AIME. In such review, verbatim abstracts up to one-tenth total content are permitted without permission.

HICROSTRUCTURAL CHARACTERISTICS OF CABOT® ALLOY NO. 214, A NEW SUPERALLOY DEVELOPED FOR APPLICATIONS IN HOSTILE, HIGH-TEMPERATURE ENVIRONMENTS

K. V. Rao and R. B. Herchenroeder

Cabot Corporation Kokomo, Indiana 46901

SUMMARY

CABOT alloy No. 214 is a wrought, Ni-Cr-Al-Fe-Y alloy developed for high-temperature applications. Because of the formation of an adherent Al $_2$ O $_3$ protective scale, formed during high-temperature exposure, the alloy exhibits the best oxidation resistance of commercially available wrought alloys to 2400°F (1315°C). This alloy also exhibits excellent carburization and chlorination resistance.

In addition to the above mentioned properties, CABOT alloy No. 214 retains good strengths at high temperatures as a result of precipitation of Ni₃Al gamma prime precipitates. The present paper deals with the mechanical properties and the microstructural features of the alloy exposed at temperatures up to $2150\,^{\circ}\mathrm{F}$ ($1180\,^{\circ}\mathrm{C}$). The thermal stability of the alloy exposed at temperatures $1800\,^{\circ}\mathrm{F}$ ($980\,^{\circ}\mathrm{C}$) for times up to 1,000 hours is presented in terms of changes in tensile properties as a function of exposure temperature and time. The results are correlated with microstructural observations made using transmission electron microscopy. A brief description of the high temperature oxidation resistance and the industrial applications of the alloy is also included.

Introduction

There is an ever increasing need for high temperature alloys which can survive service temperatures as high as 2200°F or above. Acceptable strengt' levels, oxidation resistance, and resistance to corrosive attacks by the surrounding environments such as carburizing and chlorine contaminants are frequently desirable characteristics for alloys intended for such high temperature applications.

Chromium is the basis for high-temperature oxidation resistance of most of the commercial alloys ranging from stainless steels to nickel and cobalt-base alloys. Chromium is an active and readily oxidized element which forms $\rm Cr_2O_3$ or $\rm NiCr_2O_4$ protective scales (1) at high temperature. However, at temperatures above $1000^{\circ}\rm C$, chromium tends to be oxidized to form volatile $\rm CrO_3$ (2). For this reason, the protectiveness of chromium-rich oxide scales such as $\rm Cr_2O_3$ or $\rm NiCr_2O_4$ that form on most of the conventional high-temperature alloys is limited to only short periods of time above $2000^{\circ}\rm F$ ($1090^{\circ}\rm C$).

It has been known for many years that $\mathrm{Al}_2\mathrm{O}_3$ scales formed on alloys such as Ni-15Cr-5Al are more stable and more protective than $\mathrm{Cr}_2\mathrm{O}_3$ scales against oxidation attack (3). Addition of yttrium to a Ni-15Cr-5Al alloy has been shown to improve the adherence of $\mathrm{Al}_2\mathrm{O}_3$ scales (3,4). However, the basic Ni-15Cr-5Al alloy with yttrium suffers from a serious hot workability problem. The commercialization of wrought Ni-Cr-Al-Y type alloys with good fabricability and inherent capability of producing a tenacious $\mathrm{Al}_2\mathrm{O}_3$ film at high temperatures has been successfully achieved in CABOT alloy No. 214.

CABOT alloy No. 214 is a newly developed nickel-base wrought alloy with a nominal composition of 15-17% Cr, 4-5% Al, 2-6% Fe (all wt%) and a small but effective amount of yttrium. The present paper deals with the oxidation resistance, mechanical properties and microstructural features of this alloy. The performance of CABOT alloy No. 214 was compared with Type 310 stainless steel and some of the commercial Ni-Cr-Fe alloys, such as INCONEL® alloy 601 and alloy 800H, which mainly depend on $\rm Cr_2O_3$ type scales for protection.

Experimental Procedures

The nominal compositions of the alloys investigated are listed in Table I. Oxidation test samples (approximately 0.10 to 0.15cm x 2.2cm x 2.2cm) obtained from annealed sheet materials were ground to a 120-grit surface finish. Coupons of the four alloys were simultaneously exposed at 1095°C (2000°F), 1150°C (2100°F), and 1205°C (2200°F) in flowing air with a flow rate of about 3.3 litres/minute in a 4.5cm I.D. alumina tube heated by a resistance heating furnace. The coupons were cooled to room temperature once a week (168 hours) during the exposure test. Prior to testing, the coupons were measured, cleaned, and weighed. All the samples were also turned and rotated before putting back in the furnace every week. Following exposure, coupons were weighed, electrolytically descaled, and weighed again for determination of the coupon's metal loss (mils per side of the specimen). The descaled coupons were then sectioned and examined metallographically for the depth of continuous oxide penetration. The total oxide penetration is a sum of metal loss and continuous penetration.

| | | Others Y = Present Cu = 1.0* Cu = .75* |
|---------|--|---|
| | | II 1 88. |
| TABLE I | Nominal Compositions (Wt%) of Wrought Oxidation Resistant Alloys | A1 4.5 1.35 |
| | | Mn 1.0* 2.0* 1.5* |
| | | Si. 1.5* |
| | | M |
| | | 용 |
| | | Cr 16 23 25 21 |
| | | 8 1 1 |
| | | Ni Bal 20 33 |
| | | Fe 4 14.1 Bal Bal |
| | | C .04 .10* .25* |
| | | Alloy alloy No. 214 alloy 601 Type 310 Stainless Steel alloy 800H |

* Maximum

Mechanical property measurements both in the annealed and annealed plus aged conditions were made on sheet materials exhibiting typical grain sizes in the range of ASTM 3.5 to 6.5. Thin foil specimens for transmission electron microscopy (TEM) were prepared by electropolishing in a twin jet polisher using a solution of 30% nitric acid in methanol at -20° C. The thin foils were examined using a Philips EM300 electron microscope operated at 100KV.

Results and Discussions

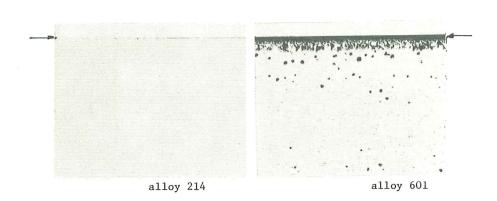
Oxidation Resistance. The results of the oxidation tests for the four alloys investigated are shown in Table II. These results clearly indicate that alloy 214 exhibits outstanding oxidation resistance of the alloys tested up to 1205°C (2200°F). In particular, at 1205°C (2200°F) oxidation resistance of alloy 214 is an order of magnitude better than alloy 601. Preliminary oxidation test results obtained at 1315°C (2400°F) for 24 hours showed that alloy 214 had an attack of less than 10 micron (0.4 mil) per side of total oxide penetration depth while other alloys suffered noticeably higher depths of penetration.

In other tests, where samples were exposed at 1150°C (2100°F) and cyled to room temperature once per day, alloy 214 exhibited 100 to 400 times less oxidation than that for alloy 601 on the basis of scaling rate, i.e., weight loss per unit of surface area for each hour of exposure.

The extent of oxidation damage at 1095°C (2000°F) and 1205°C (2200°F) for the four alloys are shown in Figures 1 and 2. These photomicrographs were obtained by viewing the cross sections of the oxidized samples after descaling. As can be seen from these micrographs, alloy 214 essentially did not suffer any subscale attack after long exposures at 1205°C (2200°F) and 1095°C (2000°F). The other alloys, namely 601, 800H and 310SS samples suffered severe oxidation attack which included metal loss, intergranular oxidation, and internal voids. X-ray diffraction analysis of the thin oxide scale on alloy 214 removed by descaling showed A1203 which is believed to impart outstanding oxidation resistance to the alloy.

A number of carburization tests performed in gas mixtures containing 5% H_2 , 5%CO, 5% CH_4 , and a balance Ar (by volume %) at temperatures up to $980\,^{\circ}\text{C}$ ($1800\,^{\circ}\text{F}$) revealed that alloy 214 is the most resistant to carburization of many commercial heat resistant alloys tested (5). Chlorination tests conducted in an oxidizing atmosphere of Ar + 20% O₂ + 2% Cl_2 , and in reducing environment of Ar + 4% H_2 + 4% HCl (6,7) at $900\,^{\circ}\text{C}$ ($1650\,^{\circ}\text{F}$) for 8 hours indicate that alloy 214 is also most resistant to attack by this corrosive media. The formation of the Al_2O_3 scale on alloy 214 has been thought to significantly minimize the chlorine-accelerated oxidation attack.

| | Total Oxide Penetration .08 2.64 1.30 7.39 | .31 5.27 4.44 8.86 | .37 4.86 5.47 8.62 | .65 7.54 10.25 13.55 |
|----------------------------|--|---|---|---|
| Static Oxidation Test Date | Total | 314 33 | 7 0, 0 | 100 |
| | Metal Loss (mils/side) .08 1.21 .97 5.39 | .15 2.36 2.97 7.52 | .15 1.59 2.24 6.63 | .22 4.42 7.95 11.27 |
| | Test Duration (Hours) 1008 1008 1008 | 1008 1008 1008 1008 | 504 504 504 504 | 1008 1008 1008 1008 |
| | Temperature °C (1095) (1095) (1095) (1095) | (1150) (1150) (1150) (1150) | (1205) (1205) (1205) (1205) | (1205) (1205) (1205) (1205) |
| | Test T %F 2000 2000 2000 2000 | 2100 2100 2100 2100 | 2200 2200 2200 2200 | 2200 2200 2200 2200 |
| | Alloy 214 601 310 Stainless Steel 800H | 214 601 310 Stainless Steel 800H | 214 601 310 Stainless Steel 800H | 214 601 310 Stainless Steel 800H |



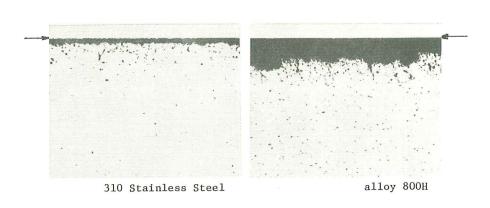


Figure 1: Optical photomicrographs (all 50%) showing comparative static oxidation attack after 1008 hr. exposure at 2000°F (1095°C). The arrow indicates position of the original surface.

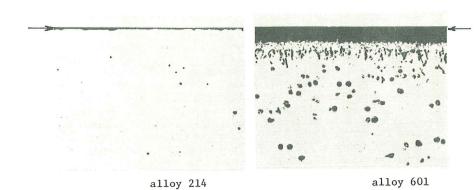






Figure 2: Optical photomicrographs (all 50X) showing comparative static oxidation attack after 1008 hr. exposure at 2200°F (1205°C) The arrow indicates position of the original surface.

CABOT alloy 214 is age-hardenable similar to the Ni-Cr-Al alloys (8). The effect of thermal aging on the room temperature tensile properties of alloy 214 is presented in Table III. A typical microstructure of the alloy in the annealed condition is shown in Figure 3a. Figure 3b shows the microstructure of alloy 214 aged at 1010°C (1850°F) for 8 hours. As can be seen from this micrograph, aging the alloy at 1850°F for 8 hours results in no significant precipitation.

Aging alloy 214 at temperatures below about 1800°F results in the formation of gamma prime precipitates. The gamma prime phase is based on the intermetallic compound Ni3Al which has the fcc Ll2 type superlattice structure. Typically overaging occurs within 100-1000 hours in the 815-980°C (1500-1800°F) temperature range. Figure 4 shows the large gamma prime precipitates present in the alloy aged at 900°C (1650°F) for 100 hours. This gamma prime is characteristic of the over-aged condition of the alloy. The data in Table III show that these coarse gamma prime precipitates formed at 900°C (1650°F) do not effectively strengthen the alloy. Aging at 800°C (1472°F) resulted in an observable increase in strengths accompanied by a slight decrease in ductility. This was attributed to relatively smaller sizes of gamma prime precipitates formed at 800°C (1472°F) aging temperatures. The typical microstructure of alloy 214 aged at 800°C (1472°F) for 100 hours is shown in Figure 5. Much finer gamma prime precipitates were developed during aging at lower temperatures such as 650°C (1200°F). This is shown for the case of 100 hours at 650°C (1200°F) in Figure 6. The finer size distribution of gamma prime precipitates were generally associated with higher tensile strength properties.

Elevated temperature strength levels of alloy 214 are much higher than alloy 601, 800H and 310 type stainless steels, at least up to 1750°F. This is illustrated in Figure 7 which shows the tensile yield strengths of the four alloys in their annealed condition (ASTM grain sizes 3-1/2 to 5). Strength levels of alloy 214 at and above 1095°C (2000°F) compare favorably with the other three alloys. The higher strength levels of alloy 214 at temperatures below about 1750°F is attributed to the gamma prime precipitation in the alloy. Thermal stability of these four heat resistant alloys, as indicated by the residual room temperature tensile elongations after aging in air for 1000 hours at 650°C (1200°F), 760°C (1400°F), 871°C (1600°F), and 981°C (1800°F) temperatures, is presented in Figure 8. This diagram illustrates the good post-aged ductility of alloy 214 after long-term high temperature exposures. This property is an important one for long-term elevated temperature service.

Conclusions

The Ni-Cr-Al-Fe-Y alloy, CABOT alloy 214 discussed in this paper, possesses superb oxidation resistance up to 1315°C (2400°F), as compared to commercially available wrought alloys. An alloy of this composition also exhibits superior corrosion resistance in carburizing and high temperature chlorine-contaminated environments. These observed properties in hostile high temperatures are attributed to the formation of thin Al_2O_3 scale on alloy 214, as compared to Cr_2O_3 and Fe_2O_3 type scales in Ni-Cr-Fe and Fe-Ni-Cr alloys.

TABLE III

Room-Temperature Tensile Properties
of Annealed and Annealed + Aged CABOT alloy 214

| Condition °F (°C) | 0.2% Offset Yield Strength | | Ultimate Tensile Strength | | Elongation in 2-in (50.8mm) | |
|-------------------------------------|-------------------------------|-----|---------------------------|------|--------------------------------|----------|
| | ksi | MPa | <u>ksi</u> | Hra | | <u>%</u> |
| Solution Annealed at 2050°F(1120°C) | 77.1 | 532 | 134 | 921 | | 40 |
| Aged 1650°F (900°C) 8 h | 77.9 | 537 | 131.8 | 909 | | 39 |
| Aged 1650°F (900°C) 32 h | 69.4 | 479 | 128.4 | 885 | | 40 |
| Aged 1650°F (900°C) 100 h | 70.1 | 483 | 127.3 | 878 | | 40 |
| Aged 1650°F (900°C) 1000 h | 61.2 | 422 | 124 | 855 | | 42 |
| Aged 1472°F (800°C) 8 h | 95.4 | 658 | 148.9 | 1027 | | 30 |
| Aged 1472°F (800°C) 32 h | 89.2 | 615 | 149.3 | 1029 | | 25 |
| Aged 1472°F (800°C) 100 h | 103.3 | 712 | 144.4 | 996 | | 16* |
| Aged 1472°F (800°C) 1000 h | 82 | 565 | 145.4 | 1002 | | 27* |
| Aged 1200°F (650°C) 1000 h | 103.8 | 715 | 157 | 1082 | | 28 |
| | | | | | | |

^{*} Qualitative results, sample broke in gage marks

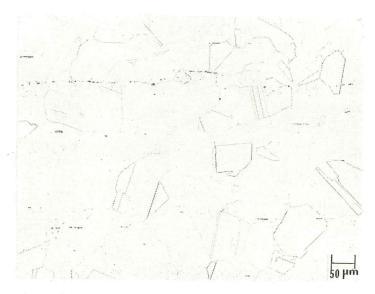


Figure 3a: Optical photomicrograph showing the microstructure of CABOT alloy No. 214 in the annealed condition



Figure 3b: TEM photograph showing essentially a single-phase structure of CABOT alloy No. 214 after aging at 1010°C for 8 hours.

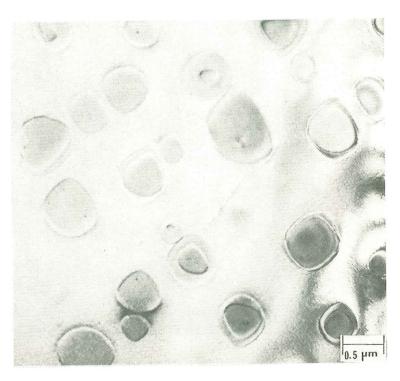


Figure 4: Bright field transmission electron micrographs showing gamma prime precipitates formed in CABOT alloy No. 214 after aging at 900°C (1650°F) for 100 hours.

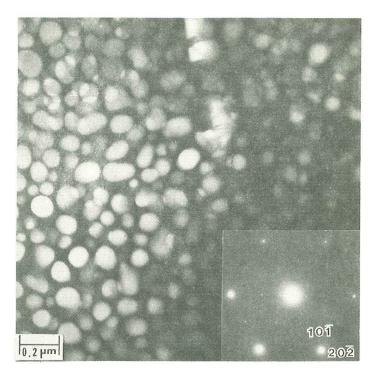


Figure 5: TEM photograph showing the typical gamma prime precipitates obtained in CABOT alloy No. 214 after aging at 800°C (1472°F) for 100 hours. Dark field micrographs taken with (10T) gamma prime reflection.



Figure 6: Dark field electron micrograph, taken with (110) gamma prime reflection, showing fine gamma prime precipitates present in CABOT alloy No. 214 aged at 650°C (1200°F) for 100 hours.

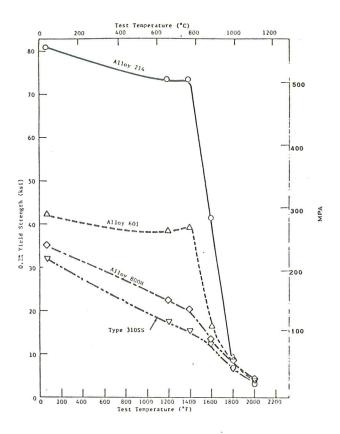


Figure 7: 0.2% offset yield strength vs. temperature of the annealed sheet material of the four alloys

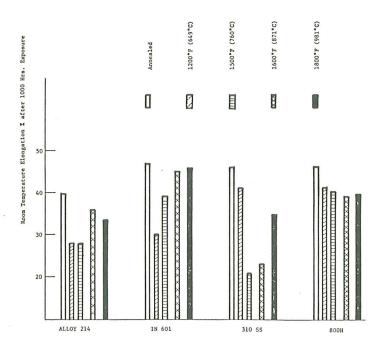


Figure 8: Room-temperature tensile elongation of the four alloys after 1000 hrs. of exposure at various temperatures

reasonably good post-aged ductility after long-term exposures to elevated temperatures. Precipitation of gamma prime phase after thermal aging and the variation in size of gamma prime precipitates appears to be the major factor responsible for the observed tensile properties and thermal stability of the alloy. The above mentioned unique properties of alloy 21 makes it a prime candidate for applications such as furnace racks, fixtures, baskets, radiant tubes, heat exchangers and recuperators, and many other components in metal and ceramic manufacturing and heat treatment industries.

Alloy 214 also exhibits much higher tensile strengths up to 1750°F and

Acknowledgments

York, p. 181 (1970).

The many contributions of the members of Cabot's Technology Department are sincerely appreciated.

REFERENCES

on the Oxidation Mechanism of Ni-20Cr", Oxidation of Metals, 2, p. 207 (1970).

2. E. A. Gulbranson and S. A. Jansson, Heterogeneous Kinetics at Elevated Temperatures, G. R. Belton and W. L. Worrell, Eds., Plenum Press, New

1. D. L. Douglass and J. S. Armijo, "The Effect of Silicon and Manganese

- A. Kumar, M. Nasrallah, and D. L. Douglass, "The Effect of Yttrium and Thorium on the Oxidation Behavior of Ni-Cr-Al Alloys", Oxidation of Metals, 8, p. 227 (1974).
- C. S. Giggins and F. S. Petit, "Oxide Scale Adherence Mechanisms and Effect of Yttrium, Oxide Particles and External Applied Loads on the Oxidation of Ni-Cr-Al and Co-Cr-Al Alloys", <u>Aerospace Research</u> <u>Laboratory Report</u>, TR-75-0234, June 1975.
- 5. R. B. Herchenroeder, G. Y. Lai and K. V. Rao, "A New, Wrought, Heat-Resistant Ni-Cr-Al-Fe-Y Alloy", Journal of Metals, 35, p. 16 November 1983.
- 6. J. M. Oh, M. J. McNallan, B. Y. Lai, and M. F. Rothman, "High Temperature Corrosion of Superalloys In Chlorine-Contaminated Environments", paper presented at the 112th AIME Annual Meeting, Atlanta, Georgia, March 1983.
- 7. S. Baranow, G. Y. Lai, M. F. Rothman, J. M. Oh, and M. J. McNallan, Unpublished results, Cabot Corporation, Kokomo, Indiana.
- 8. A. Taylor and R. W. Floyd, "The Constitution of Nickel-Rich Alloys of the Nickel-Chromium-Aluminum System", <u>Journal of Institute of Metals</u>, 451, p. 81 (1952).