EFFECTS OF COBALT ON THE HOT WORKABILITY

OF NICKEL-BASE SUPERALLOYS

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Summary

It is found that reducing the cobalt content in representative wrought nickel-base superalloys can result in an increase in both the γ' solvus temperature and the solvus temperature of carbides which precipitate as continuous and brittle intergranular networks. It is further found that these solvus temperatures border the hot working temperature range for the alloy. The standard working range for the alloy with 14% cobalt is between these However, removing the cobalt raises the transition two phase boundaries. temperatures so the standard working range now lies below the carbide It is shown that the resulting stringers of carbide can either hamper heat treatment and diminish mechanical properties (in low carbon alloys) or eliminate workability altogether (in alloys containing more carbon). Thermomechanical processing modifications reflecting the effect of cobalt on the γ' and carbide solvi are shown to restore the workability and the properties of low and no cobalt alloys. The results of this study refute the often accepted wisdom that the strategic element cobalt is necessary for hot workability and for properties.

Introduction

As part of the NASA Conservation of Strategic Aerospace Materials (COSAM) program [1,2], we are studying the role of cobalt on various nickel-base superalloys with the aim of reducing the consumption of this strategic element in critical aerospace applications. Superalloys (predominantly the wrought nickel-base alloys containing between 10 and 20% Co) consume roughly 40% of the cobalt in the United States [3].

Prior to research [4-12] spurred by the cobalt supply crisis at the end of the 1970's, much of the information concerning the role of cobalt in nickel-base superalloys had originated in Heslop's 1964 paper [13]. He reported that a 17% cobalt addition to Nimonic 80A (an early γ' strengthened superalloy) markedly improves the stress rupture resistance of this alloy. The Nimonic alloys developed after this cobalt containing version of Nimonic 80A contain between 13 and 20% cobalt. Similarly, their American counterparts—Waspaloy, the Udimet alloys, the Rene alloys, and the Mar M alloys, for example—all contain from 8 to 19% cobalt [14]. The effects of cobalt on the structure and properties at service temperatures were recently studied in some detail in many of these alloys [4-12], and it was found that the role of cobalt is not as critical as believed in terms of enhancing mechanical properties in this temperature range.

Heslop [13] also noted that the hot ductility (or workability) of high strength superalloys, based on studies of Nimonic 115 type alloys, increased uniformly with alloy cobalt content. This conclusion was supported in the paper by a sketch without data points showing ductility (twists to failure on a torsion Plastometer) in the hot working temperature range increasing linearly with cobalt content. This information has been cited repeatedly as evidence for cobalt being necessary to enhance hot workability of superalloys [3,15,16] and has become part of the 'common wisdom' concerning cobalt. In this paper we report on our recent study of this issue. In so doing, we will show that wrought superalloys are workable even in the absence of cobalt. For the study we chose to concentrate on that very same Nimonic 115 alloy originally studied by Heslop. These results, and results from other alloys, are shown to be generally applicable to the high strength (high γ' fraction) nickel-base superalloys. Some information of the effect of hot working procedure on mechanical properties of Nimonic 115 and Udimet 700 will also be presented.

Experimental Procedure

Four 68 kg. (150 lbs.) heats of alloys based on the Nimonic 115 composition with varying amounts of nickel substituted for the nominal 14% cobalt were prepared for this study, Table I. Also given in Table I are chemistries of alloys referred to later during discussion. Note that with the exception of the systematic nickel/cobalt substitutions, all other alloy contents are constant in each of the systems studied. These alloys were vacuum induction melted (VIM) and cast at Special Metals Corporation of New Hartford, New York, in a 227 kg (500 lb.) capacity furnace. To refine the grains of the cast microstructure these ingots were then vacuum arc remelted (VAR) into 150 mm (6 inch) diameter ingots. Since these alloys were produced by and bought from Special Metals Corporation, the designation Nimonic 115 alloy should, strictly speaking, be Udimet 115.

Primary breakdown of the cast structure in superalloys is a most difficult processing step. Planes of weakness [17,18] are a result of the casting segregation combined with the columnar grains. This structure must

be deformed and homogenized before shape forming (for example, hot forging) can proceed [19-22]. Simulation tests, whether they are tensile, torsion or compression, cannot reproduce the conditions in the primary deformation stage where the failure mode is predominantly edge cracking or centerline bursting [18,23]. For this reason we examined the hot workability by direct observation of hot rolled ingots. Standard mill procedures for Nimonic 115 (or Udimet 115) were followed as to the reduction per pass, and the hold times for the thermal processing steps—homogenization, overaging, and reheat between deformation passes. Initially, the standard temperatures prescribed for these processing steps were used. Modifications in these processing temperatures are discussed below.

Table I. Alloy Compositions (in weight %)

Heat #	Ni	Co	Ст	Ti	A1	Мо	С	В
Nimonic 115								
D5-2280	Bal.	13.8	14.6	3.95	4.91	3.52	.159	.0165
D5-2281	Bal.	10.0	14.6	3.87	4.75	3.50	.164	.0175
D5-2282	Bal.	5.2	14.4	3.97	4.88	3.50	.149	.0175
D5-2283	Bal.	<0.2	14.4	3.91	4.81	3.45	.141	.0178
D5-2520	Bal.	⟨0.1	14.4	4.03	4.93	3.48	.144	.0183
D5-2521	Bal.	5.1	14.5	3.94	4.90	3,53	.153	.0177
D5-2522	Bal.	10.0	14.5	3.93	4.97	3.55	.157	.0180
D5-2523	Bal.	15.0	14.6	3.99	4.88	3.54	.151	.0175
Udimet 700 [9	,10]							
D5-1884	Bal.	<0.1	15.1	3.46	4.12	5.00	.06	.025
D5-1885	Bal.	4.3	15.1	3.55	4.14	4.90	.07	.024
D5-1886	Bal.	8.6	15.0	3.51	4.05	5.05	.06	.022
D5-1932	Bai.	12.8	14.7	3.61	4.10	5.00	.06	.023
D5-1933	Bal.	17.0	14.9	3.60	4.08	5.03	.06	.028
D1-3073	Bal.	0.2	15.0	3.54	4.00	4.70	.07	.032
D1-3074	Bal.	4.9	14.4	3.57	4.02	4.77	.06	.030
Udimet 720 [5	,25]							
D5-2284	Bal.	14.8	17.9	4.95	2.46	3.09	.037	.031
D5-2285	Bal.	7.5	17.8	5.03	2.52	3.11	.031	.031
D5-2286	Bal.	<0.2	17.6	4.99	2.48	3.04	.036	.030
	All Udime	et 720	alloys	contain	1.2% W			
IN 738 [25]								
D1-2944	Bal.	8.4	16.2	3.37	3.46	1.64	.096	.008
D1-2945	Bal.	0.4	16.1	3.33	3.47	1,62	.097	.009
D1-2946	Bal.	4.2		3.35	3.49	1.64	.095	.008
	A11 IN 7			ain 2.49	6 W. 1.	б% Та.	and 0.7	4% Nb

For all alloys: Fe<0.18%, S<.003%, P<.005%, 0,N<12 ppm

Following standard practice for pre-rolling treatments, the VIM/VAR ingots were homogenized for 24 hours at 1204°C (2200°F). After a furnace cool, the alloys were overaged for 10 hours at 1107°C (2025°F) to precipitate and coarsen a small fraction of the γ' phase. These precipitates pin grain boundaries to prevent abnormal growth during hot working. The ingot surfaces were ground to remove any defects and then the ingots were sealed in 6 mm (1/4 inch) thick mild steel cans to prevent surface chilling during rolling.

The canned ingots were rolled at 1093°C (2000°F) to a final target of 19 mm (3/4 inch) round rods. The standard procedure for this material calls for squaring passes until the ingot is reduced to a roughly 40 mm (1.6 inch) round corner square (RCS) bar. The reheating time between passes was 30 minutes to equilibrate the piece to the rolling temperature. The steel jacket was then removed from the piece and it was rolled to 20 mm (.8 inch) RCS bar. The finishing passes first reduce the bar to an oblong cross section 15x25 mm and then to the final 19 mm round. During these operations the reheat times were reduced to 15 minutes since the smaller cross sections equilibrate more rapidly. Sections of the low cobalt alloys were also over-

aged and rolled at 1176°C (2150°F) into 13x62 mm (0.5x2.5 inch) plate, see below for details on these rolling experiments.

Optical and SEM metallography were used to characterize the material at the various stages of processing (i.e., after casting, homogenizing, overaging and hot working). Particular attention was paid to the chemistry and morphology of the carbides after each step of thermal mechanical processing. The SEM specimens were electropolished in a 20% $\rm H_2SO_4$ -methanol solution. These specimens were then etched in either a 10% HC1-methanol solution to reveal the carbide structure or an aqueous 1% (NH₄)₂SO₄-1% citric acid solution to reveal the γ' structure. With these etchants the carbides are distinguished by appearance in the SEM. MC carbides appear chunky and are uniformly dispersed and the $\rm M_{23}C_6$ carbides are found only at heterogeneities (particularly the grain boundaries). The chemistry of the carbides were also determined using an energy dispersive X-ray analysis (EDS) unit in a JEOL JEM-100CX analytical electron microscope. In these alloys carbides with high chromium content are $\rm Cr_{23}C_6$ type and carbides with high titanium content are TiC type, as confirmed by X-ray diffraction of extracted carbides [24].

The γ' solvus of each composition was determined using a Perkin-Elmer Differential Thermal Analyzer (DTA) with a temperature accuracy of approximately 5°C. The $\mathrm{Cr}_{23}\mathrm{C}_6$ carbide solvus for each alloy composition was determined metallographically on homogenized cast material aged for 24 hours at various temperatures followed by a water quench, see next section for details on this characterization.

Results and Discussion

Removing cobalt from Nimonic 115 causes a drastic reduction of the primary workability at the standard rolling temperature of 1093° C (2000° F). The alloys with 0 and 5% cobalt failed during the initial rolling pass after a reduction in area of less than 10%, Fig. 1. In contrast, the alloys containing 10 and 14% (nominal Nimonic 115) cobalt were successfully rolled to the targeted 19 mm (3/4 inch) rods—a total reduction in area of greater than 98%. The reduced cobalt alloys fractured in a brittle manner when rolled at the standard hot working temperature. The microstructure of these materials, seen in Fig. 2, includes $Cr_{23}C_6$ carbides forming a continuous network at the grain boundaries. Fracture analysis shows that the cracks propagate along these intergranular carbides, Fig. 3.

In Udimet 700 [9,10] and Udimet 720 [5,25], we found that reducing the cobalt content also resulted in the formation of stringers of y' and carbides or borides, Fig. 4. Although these alloys were rolled successfully (since these alloys contain less carbon and therefore fewer stringers), the resulting wrought structures interfered with heat treatment for optimal properties. Cobalt-free Udimet 700, after a full solution treatment (used in creep resistant applications such as turbine blades), was at least equal to the standard alloy in tensile, creep and stress rupture strengths [9,10]. However, the same alloy did not respond to the standard sub-solvus heat treatment used for lower temperature disk applications where fatigue resistance and tensile strength require a finer grain size. The γ' incorporated in the stringers was unavailable for its critical role as a dispersed particle strengthener. The low cobalt alloys had effectively less γ' and therefore lower strength. The stress rupture resistance of Udimet 700 [9,10] after standard thermomechanical processing (TMP) dramatically shows this effect, lower curve in Fig. 5. Low cobalt Udimet 720 [5,25] could not be heat treated -- the stringers would not dissolve below the incipient melting point.



Fig. 1. Alloy D5-2283 with 0% cobalt after attempting to hot roll the alloy using the standard procedure for Nimonic 115.

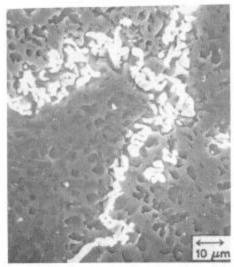


Fig. 2. SEM micrograph showing the continuous grain boundary carbides in the alloy in Fig. 1.

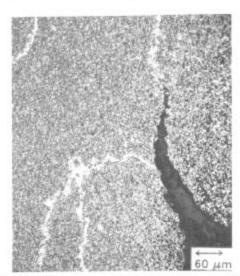


Fig. 3. Micrograph of secondary cracks near the fracture surface in the 0% Co slloy in Fig. 1.

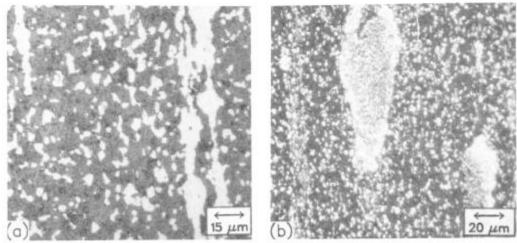


Fig. 4. Micrographs of stringers cobalt-free Udimet 700 (a) and Udimet 720 (b) resulting from using standard processing temperatures.

Since both the TiC and the $Cr_{23}C_6$ carbides contain only traces of cobalt [24], it is unlikely that cobalt directly affects the stability of those carbides. However, cobalt has been shown to affect the γ' solvus of nickel-base superalloys [3-14], and it therefore may affect the solvus between $Cr_{23}C_6$ and TiC carbides by altering the activities in the γ matrix of such carbide and γ' forming elements as titanium. Differential thermal analysis (DTA) on the alloys reveals that removing cobalt indeed increases the γ' solvus of the Nimonic 115 type alloys from 1178°C in the standard alloy to 1228°C in the cobalt-free version, see Fig. 6. This effect also occurs in Udimet 700, Udimet 720, IN 738, MERL 76, and MarM 247 [5-12] so the results of the current study on Nimonic 115 may perhaps be generally applicable to high strength superalloys.

To determine the precipitating carbide solvus and simulate the thermal processing prior to deformation, cast material from each of the four Nimonic 115 heats were solutioned now at 1240°C (2264°F) and overaged at various lower temperatures for 24 hours. The cast microstructures are similar except that a quantity of very fine $\mathrm{Cr}_{23}\mathrm{C}_6$ carbides had precipitated in the 0 and 5.2% cobalt alloys during cooling, Fig. 7. Homogenizing the alloys at 1240°C dissolves these carbides so the microstructures of all four alloys were very similar at this point—all γ' is solutioned and the only precipitates are the primary TiC carbides and a few borides, Fig. 7, center.

During next processing step, overaging at 1107°C (2025°F), the carbide stringers form in the low cobalt alloys but not in standard Nimonic 115, see Fig. 7. In the standard alloy, only a small amount of discrete γ' and TiC particles are found at the grain boundaries. However, in the cobalt-free alloy (and similarly the alloy with 5.2% cobalt) a continuous film of precipitated Cr₂₃C₆ is the striking microstructural feature. Microstructures of the cobalt-free alloy at the modified overaging temperatures chosen for rolling are shown in Fig. 8. Note the marked similarity of the cobalt-free alloy overaged at 1175°C and the standard alloy overaged at 1107°C. Overaging at 1150°C produces a very coarse precipitation of the carbides at various locations within the cobalt-free alloy. These heavy precipitates are not continuous, indicating that 1150°C is very near the carbide solvus temperature—it may be above or below depending on local chemistry.

This ${\rm Cr_{2\,3}C_6}$ solvus as a function of cobalt content in the homogenized cast alloys, as determined metallographically, is included in Fig. 6. Note that this carbide solvus follows the same trend as the γ' solvus and, again, that the pre-rolling overage temperature 1107°C (2025°F) is above the ${\rm Cr_{2\,3}C_6}$ carbide solvus for the alloys with greater than 10% cobalt and is below the solvus for the alloys with lower cobalt contents. Not surprisingly, the 10 and 14% cobalt containing alloys rolled successfully and the alloys containing 5.2 and 0% cobalt did not. Conversely, the hot workability of the low cobalt alloys should be enhanced if the overage and rolling temperatures are raised above the ${\rm Cr_{2\,3}C_6}$ solvus in Fig. 6.

This premise was confirmed experimentally by removing a 50x50x100mm (2x2x4 inch) section of the piece shown in Fig. 1 and a similar section of the 5.2% cobalt alloy and attempting to re-work this material. Since the $M_{23}C_6$ carbide reaction is generally reversible in superalloys, the two sections were homogenized for 24 hours at $1204^{\circ}C$ ($2200^{\circ}F$) to dissolve these carbides and overaged at $1177^{\circ}C$. Both the 0 and the 5.2% cobalt alloys were successfully rolled at $1177^{\circ}C$ to 13 mm (1/2 inch) thick plates after this thermal treatment.

Similar alloys (D5-2520 and D5-2521 in Table I) were homogenized at 1204°C, overaged at 1163°C (2125°F), and rolled at 1149°C (2100°F) in the

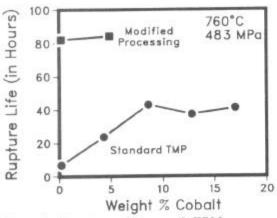


Fig. 5. Rupture lives of U700 as a function of cobalt content at 760C and 483 MPa.

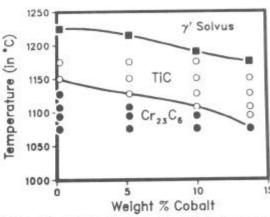


Fig. 6. Plot of the γ^* solvus and the carbide transformation temperatures in N115 as a function of cobalt content.

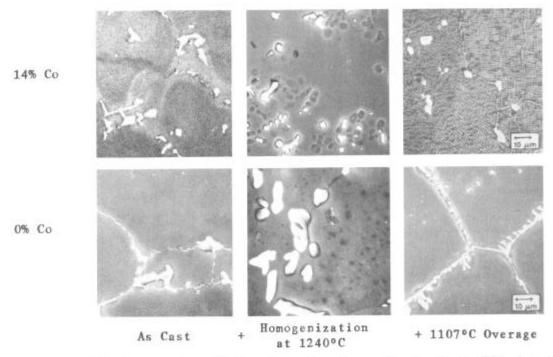


Fig. 7. SEM micrographs of the microstructures in standard N115 (top) and and the cobalt-free alloy (bottom) formed during thermal treatments prior to hot working.

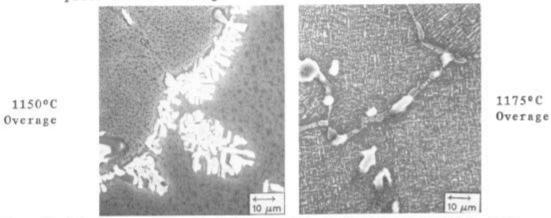


Fig. 8. Cobalt-free Nimonic 115 overaged at 1150°C and 1175°C. Note that the discontinuous carbide precipitates formed at 1150°C are absent at 1175°C.

production mill at Special Metals Corporation. The alloy with 5% cobalt (D5-2521) was successfully worked from the 150 mm (6 inch) VAR ingot to 19 mm bar with very little reconditioning with a yield that was at least as much as the standard alloy (D5-2523). Conditioning losses for the cobalt-free alloy were high (sections with a good deal of surface cracking must be removed). In order to reduce any material successfully from the 22 mm RCS to 19 mm round bar, the rolling temperature was raised to 1177° C (2150° F). The resulting microstructure in this alloy suffers from the same deficiency of strengthening γ' as Udimet 700 in our earlier study [10] and its mechanical properties decrease accordingly, Fig. 9.

Micrographs of the cobalt-free and the standard Nimonic 115 wrought alloys are shown in Fig. 10. Only slight differences in the γ' and in the grain sizes distinguish the standard alloy worked at 1094° C from the cobalt-free version overaged and worked at 1177° C. (Compare to the stringered microstructure of the cobalt-free alloy worked at 1149° C.) The fine grain size and γ' distribution in the materials in Figs. 10a and 10b are typical of extruded or rolled billet. Material with this structure is amenable to additional working or heat treatment. These wrought alloys were successfully heat treated with the solution temperature also adjusted to the increased γ' solvus of the low cobalt alloys. The mechanical properties of these alloys are also shown in Fig. 9, indicating that the effects of cobalt can be removed with proper thermo-mechanical processing.

To further test the effect of the transformation temperatures (or cobalt content) on the thermo-mechanical processing, we worked two low cobalt heats of Udimet 700 [25] at temperatures prescribed by its γ' and carbide solvi. The standard practice temperatures used in this study for Udimet 700 call for homogenization at 1177°C (2150°F), overaging at 1107°C (2025°F) and rolling at 1094°C (2000°F). Heat D1-3073 (0% Co) was homogenized at 1204°C (2200°F), overaged at 1163°C (2125°F), and rolled at 1149°C (2100°F). For heat D1-3074 (5% Co) the temperatures were, respectively, 1191, 1149, and 1135°C (2175, 2100, and 2075°F). The resulting wrought microstructures did not contain the stringers and were markedly similar to the standard alloy, Fig. 11. Not surprisingly, the stress rupture resistance of these alloys (often cited as the mechanical property most sensitive to cobalt content) improved substantially, see top curve in Fig. 5.

It may be fortuitous that both these reduced cobalt alloys exceed the stress rupture resistance of the standard alloy. However, the results clearly demonstrate that the strategic element, cobalt, is not an essential alloying element in the alloy Udimet 700. The invariance in the properties of Nimonic 115 with cobalt content indicates that the cobalt currently used in this whole class of alloys may be unnecessary and possibly detrimental (as in the case of oxidation and hot corrosion resistance [5]).

We emphasize the importance of the phase transformation temperatures in and around the hot working temperatures of nickel-base superalloys. In this study we used cobalt as a means of altering these temperatures without intrinsically affecting the alloy. Cobalt's effect on the γ ' solvus has been known for some time—it can be seen directly from the ternary Ni-Al-Co phase diagram and pseudo-binary Ni-Al diagrams [9,13,26]. Cobalt does not form a Co₃Al phase equivalent to Ni₃Al, so its presence in γ ' would be expected to de-stabilize (or lower the solvus of) that phase. We believe a better understanding of the effect of cobalt (and the γ ' solvus) on the carbide phases of superalloys has developed from this study. Consequently, this understanding resulted in the redesign in of TMP procedures which enhanced the workability and improved the mechanical properties of low or no cobalt superalloys.

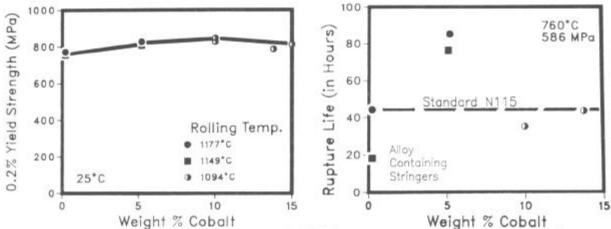


Fig. 9. Room temperature tensile and 760°C stress rupture properties of Nimonic 115 as a function of cobalt content. Notice the difference in rupture lives of the 0% Co alloy when worked at 1177°C compared with the same cobalt content alloy worked at 1149°C.

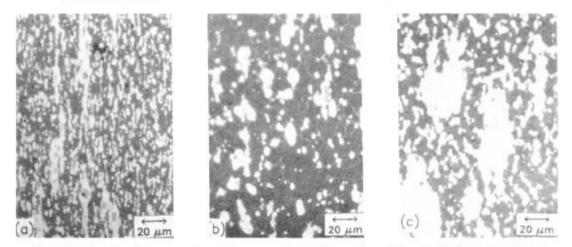


Fig. 10. Optical micrographs of as rolled Nimonic 115: (a) the standard alloy worked at 1094°C, (b) the cobalt-free alloy worked at 1177°C and (c) worked at 1149°C. The light areas in the micrographs are γ' and the large light areas in (c) are the γ' /carbide stringers.

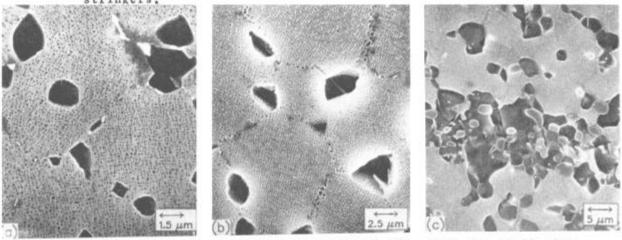


Fig. 11. SEM micrographs of heat treated Udimet 700. Note the similarity between the microstructure of the standard alloy (a) worked at 1094°C and the cobalt-free alloy (b) worked at 1149°C. In contrast is the cobalt-free alloy worked at 1094°C (c) filled with stringers of γ' and carbides.

Finally, this link between the γ' solvus and the transition or solvus temperature between the TiC and the Cr_{23}C_6 carbides can be easily rationalized. The γ' phase with its Ni₃(A1,Ti) base composition competes with the TiC carbide for titanium. Gamma prime precipitation results in the γ matrix being depleted of titanium and enriched with chromium, which is rejected by the γ' [10,27,28], thus favoring the reaction

$$\frac{23}{6} [Cr] + TiC \longrightarrow \frac{1}{6} Cr_{23}C_6 + [Ti]$$

where [X] represents element X in solution in the γ matrix. Such an explanation is consistent with the results of Collins [29] showing the transformation of MC carbides to $M_{23}C_6$ at temperatures below the γ' solvus for a for a range of alloys. The connection between the γ' solvus and $TiC/Cr_{23}C_6$ transformation temperature is then due to the temperature dependence of the relative titanium and chromium activities. Cobalt is not a component of either type of carbide and it is only a minor component of γ' . However, it does affect the γ' solvus, and hence, the activity of titanium and chromium in the matrix resulting in the temperature dependence of the carbide solvus. For this reason a high γ' solvus would result in the $Cr_{23}C_6$ being stable to higher temperatures in the low cobalt alloys.

Concluding Remarks

The effects of cobalt on the hot workability of Nimonic 115 can be summarized as follows:

- 1) Cobalt lowers the γ' solvus, which in turn, lowers the Cr_{23}C_6 carbide solvus.
- 2) The hot working range is bracketed by these transformation temperatures, since below the $\mathrm{Cr}_{23}\mathrm{C}_6$ solvus, brittle carbide networks deleteriously affect workability and above the γ' solvus abnormal grain growth can occur. This conclusion may be generally applicable to superalloys.
- 3) Accordingly, removing cobalt from Nimonic 115 (or similar alloy) necessitates an increase in the hot working temperature of the alloy in order to recover hot workability.
- 4) The mechanical property losses in this alloy and in Udimet 700 with the reduction of cobalt are shown to result from the change in the carbide transformation temperature and can be remedied with the self-same changes in processing temperatures which had restored the lost workability.

We emphasize that similar studies on the wrought alloys Udimet 700 [5,25] and Udimet 720 [25] also show that both γ' solvus and the stringered carbide solvus temperatures appear to increase as cobalt is removed from the alloy. In cobalt-free versions of these alloys, the stringers resulted in complications in the normal heat treatment which were remedied with the appropriate thermomechanical processing. Accordingly, contrary to popular belief, cobalt is not essential for hot workability nor is it essential for mechanical strength.

An interesting application of these results may be as a possible alternative to the expensive powder metallurgy route for processing high

strength turbine disk alloys. The current P/M alloys-Astroloy, IN 100, MERL 76, Rene 95 and AF 115-all contain between 15 and 20% cobalt and as ingots are currently too strong to conventionally hot work just below their γ' solvi. Based on our results, it is fair to assume that removing cobalt would raise the γ' solvus in these alloys roughly 50°C. This may correspondingly allow for the hot working of these alloys at higher temperatures, but still below their new γ' solvi, where the alloys would be more deformable.

A preliminary study of this proposal on a small (14 kg.) heat of IN 738, Table I, showed that roughly a 40% reduction in the peak rolling resistance (a function of hot working flow stress) resulted from the 55°C increase in working temperature allowed by the removal of the cobalt from the standard alloy containing 8.5% cobalt, Fig. 12. These stresses are calculated from the current drawn by a constant speed rolling mill and the strains are determined by the reduction per pass. This and other work on the hot workability of ingots of a cobalt-free MERL-76 type superalloy is in progress.

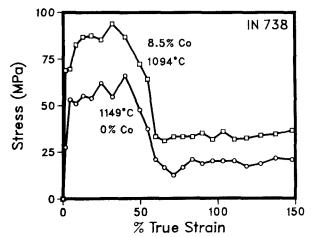


Fig. 12. Plot of the resistance to rolling of IN 738 [25].

Acknowledgements

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