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The effect of residuals on the elevated temperature properties of some creep resistant steels

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The effect of residuals and other deliberate minor additions on the elevated temperature properties of austenitic, CrMo and CrMoV steels is reviewed and those that affect these properties are identified. The elements boron, molybdenum, nitrogen and phosphorus in austenitic steels all increased creep rupture life although only boron and molybdenum were beneficial to rupture ductility. In the ferritic steels the embrittling elements antimony, arsenic, phosphorus and tin were considered together with aluminium, copper, silicon, titanium and boron. It is apparent that the effect of an individual element on creep rupture properties is dependent on the other elements present. However, in a 1 %CrMoVTiB steel additions of copper plus nickel and arsenic plus tin decrease rupture life although only the latter two reduce ductility. Similarly, in a $2\frac{1}{4}$ %Cr1 %Mo steel arsenic has a detrimental effect on ductility and tin and phosphorus have been identified as segregating to prior austenite grain boundaries. In contrast, silicon in a $2\frac{1}{4}$ %Cr1 %Mo steel can improve ductility. Aluminium can improve both the creep life and ductility of 1 %CrMoVTiB steels, as can boron in the presence of titanium.

1. INTRODUCTION

With the increasing levels of trace elements being found in steels, emphasis in recent years has been put on the effect of residual elements on the high temperature properties of creep resisting steels. In austenitic steels the effects of trace elements such as boron, nitrogen and phosphorus have been studied in an attempt to optimize creep properties, reduce scatter in creep data of a particular grade of steel and to reduce the level of expensive alloying additions such as nickel and molybdenum. For ferritic steels the problem is different. Here, although cheap steels with adequate high temperature strength are available, a number of these suffer low ductility associated with intergranular fracture. Poor ductility has been associated with high residual contents in these steels and there is evidence to suggest that residual elements such as arsenic, antimony, phosphorus and tin which have been identified as segregating to grain boundaries, causing temper embrittlement, may be responsible for intergranular low ductility creep fracture.

This paper reviews work carried out at the authors' laboratories with reference to previously published work on both austenitic and ferritic steel to identify the elements causing major changes in rupture properties and the mechanisms by which these changes occur.

2. EFFECT OF RESIDUALS IN AUSTENITIC STEELS

2.1. *Effect of boron*

Although boron was initially added to austenitic steels to improve hot workability, it was subsequently found that small additions of boron increased creep strength and ductility (Henry

& Philibert 1970; Keown 1973; Matsuo *et al.* 1973). The effects of boron additions between 40 and 50 μg/g on the rupture life of research casts of type 304, type 316, type 347 and 16 %Cr10 %Ni6 %MnNbV austenitic steels are illustrated in figure 1 (N. G. Needham & F. R. Beckitt 1978, unpublished). Although there is little effect of boron on the type 304 steel, the more complex precipitation-hardened type 347 and 16 %Cr10 %Ni6 %MnNbV steels show a large increase in rupture life, particularly at the lower stresses. Rupture ductility is unaltered by boron additions despite the increase in strength, except for the type 316 steel where increases of up to 30 % in ductility were found. This increase in ductility was associated with a reduction in the amount of intergranular cavitation at fracture.

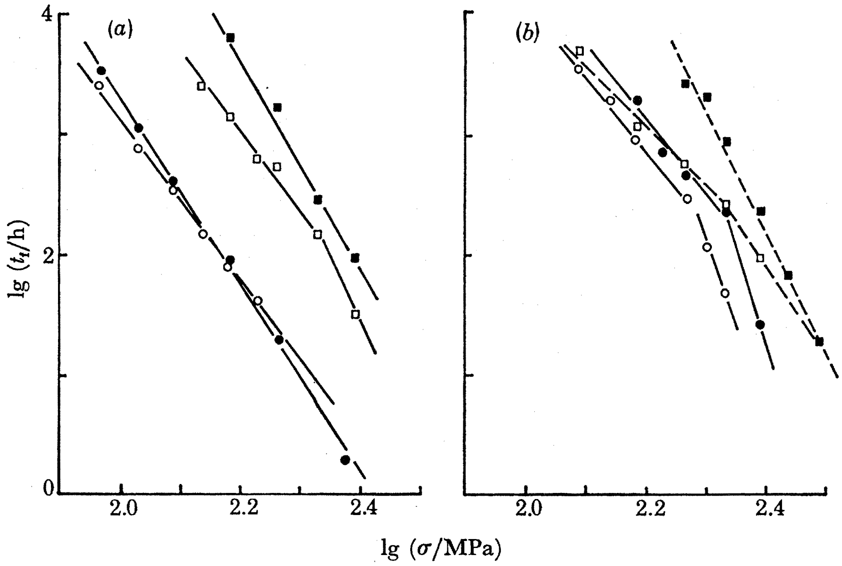

FIGURE 1. Relation between time to fracture, t_f, and applied stress, σ, at 650 °C for (*a*) a type 304 steel with boron contents of 13 μg/g (○) and 52 μg/g (●) and a type 347 steel with boron contents of 13 μg/g (□) and 47 μg/g (■) and (*b*) a type 316 steel with boron contents of 14 μg/g (○) and 44 μg/g (●) and a 16 %Cr10 %Ni6 %MnNbV steel with boron contents of 11 μg/g (□) and 54 μg/g (■).

High sensitivity creep tests show the same trends for secondary creep rate as was found for rupture life (see figure 2). The effect of boron increases, relative to the steel not containing boron, as the creep strength is increased. Over the stress range used, the stress dependence follows Norton's Law with a stress index of between 5.0 and 9.7, except for the low stress data of the type 316 and 16 %Cr10 %Ni6 %MnNbV steels where there is some indication of a decrease in stress index. Although boron reduces the secondary creep rate in the high stress dislocation creep régime, it has no significant effect on the stress index or the controlling deformation mechanism. The increase in creep strength associated with the boron containing steels is consistent with electron metallographic studies which revealed more numerous and finer carbides, which were more evenly distributed in the matrix of the boron containing steels. The change in stress index at low stress for the type 316 and 16 %Cr10 %Ni6 %MnNbV steels is consistent with other data (Beckitt & Gladman 1978) showing a change from a dislocation creep mechanism to a diffusion creep mechanism.

Similar improvements in rupture life have been observed in commercial casts of type 316 steels. Regression analysis of the 10000 h creep rupture stress shows that of the elements present boron has the strongest effect on creep rupture life (see table 1).

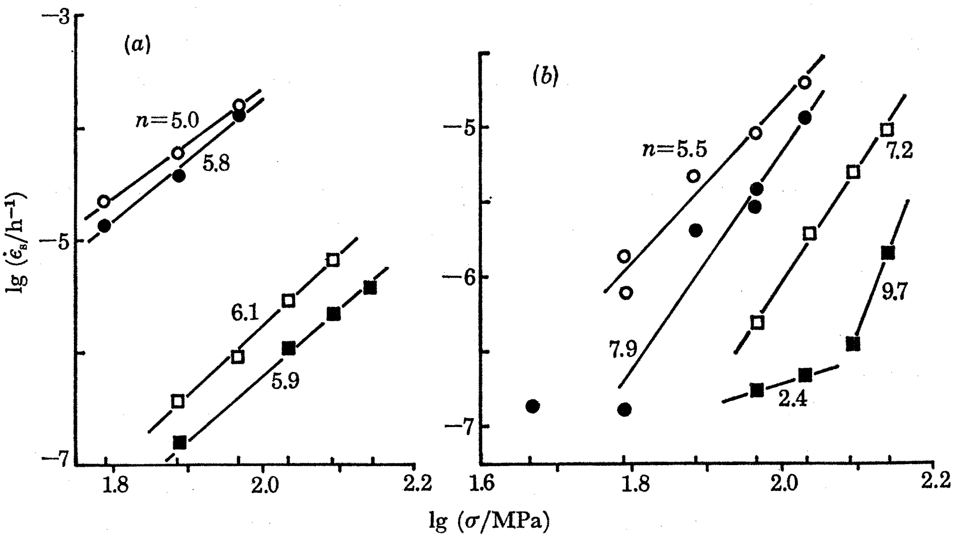

FIGURE 2. The stress dependence of the secondary creep rate, $\dot{\epsilon}_s$, at 650 °C for (*a*) a type 304 steel with boron contents of 13 μg/g (○) and 52 μg/g (●) and a type 347 steel with boron contents of 13 μg/g (□) and 47 μg/g (■) and (*b*) a type 316 steel with boron contents of 14 μg/g (○) and 44 μg/g (●) and a 16 %Cr10 %Ni6 %MnNbV steel with boron contents of 11 μg/g (□) and 54 μg/g (■).

These results are consistent with previous work on the effect of boron in similar steels (Henry & Philibert 1970; Keown 1973; Matsuo *et al.* 1973). They support the suggestion (Henry & Philibert 1970; Matsuo *et al.* 1973) that boron increases the creep strength in the dislocation creep régime by refinement and stabilization of the carbide precipitates and that it hinders intergranular cavity growth, thus increasing the rupture life.

TABLE 1. FACTORS AFFECTING RUPTURE LIFE OF TYPE 304 AND TYPE 316 STEELS

(Chemical symbols indicate concentrations of elements in percentages by mass.)

steel	temperature/°C	regression equation derived
type 316	650	$R_s = 173.8 + 7243\,\mathrm{B} + 961.1\,\mathrm{N} + 1145\,\mathrm{S} - 7.5\,\mathrm{Cr}$
	700	$R_s = 123.8 + 2930\,\mathrm{B} + 336.5\,\mathrm{N} - 4.4\,\mathrm{Cr}$
type 304	550	$R_s = 171 + 101.1\,\mathrm{Mo}$
	650	$R_s = 90.81 + 115\,\mathrm{Mo} + 498.5\,\mathrm{N}$

R_s = stress to rupture in 10000 h (megapascals).

2.2. *Effect of nitrogen*

Nitrogen additions to commercial austenitic steels have been used for a number of years and have led to the development and production of high nitrogen grades of type 304, type 316 and type 347 steels (British Steel Corporation 1973). In these steels (British Steel Corporation 1973; Goodell *et al.* 1967; Matsuo *et al.* 1973; Smith *et al.* 1959), nitrogen increases rupture life but decreases both rupture ductility and secondary creep rate. The effect of nitrogen on rupture life of type 304 and type 316 commercial casts is again illustrated by the regression analysis on the 10000 h rupture stress for these steels (see table 1). Both steels show a strong dependence on the nitrogen content, particularly at 650 °C. Further, a comparison of the results at 650 °C confirms the finding (Kawabe *et al.* 1968) that the increase in rupture life is greater and reduction in ductility smaller in molybdenum bearing steels. Although there appears to be a limiting amount of nitrogen which can be added, above which little improvement in rupture life occurs (Goodell *et al.* 1967; Okamoto *et al.* 1963), it is apparent that nitrogen has essentially the same effect as carbon (Goodell *et al.* 1967; Keown & Pickering 1972). This is particularly so in niobium stabilized steels where the rupture life is dependent on the niobium to carbon plus nitrogen ratio (Keown & Pickering 1972).

The effect of nitrogen on rupture properties in niobium stabilized steels emphasizes the role that nitrogen has on structure and strength. At the stoichiometric composition of the ratio of niobium to carbon plus nitrogen the maximum amount of fine niobium carbonitride is formed and the strength of the matrix and rupture life is maximized. In a type 316 steel (Matsuo *et al.* 1973), nitrogen refines the precipitate size and as rupture life is inversely related to the precipitate size in this steel, nitrogen increases the rupture life. Further, nitrogen suppresses the formation of ferrite and sigma phase (Monkman *et al.* 1956) which are both associated with enhanced cavitation and reduced rupture life. However, in an 18 %Cr7 %Ni steel, nitrogen levels above 0.32 % by mass result in the formation of lamellar nitrides and a decrease in rupture strength results (Okamoto *et al.* 1963). This reduction in rupture strength occurs at nitrogen levels above those used in commercial grades where 0.25 % nitrogen (by mass) is the upper limit (British Steel Corporation 1973).

2.3. *Effect of molybdenum*

Although primarily added to increase high temperature corrosion resistance, molybdenum improves high temperature creep rupture properties. Recently, however, there has been an increase in residual molybdenum in commercial type 304 austenitic steels which has resulted in better rupture properties than those accepted for this grade. Regression analysis on a number of commercial casts with molybdenum levels from 0.03 to 0.71 % by mass showed the strong dependence of the 10000 h rupture stress on molybdenum at both 550 and 650 °C (see table 1). Rupture ductility was unaffected. Similarly, additions of 0.7 % molybdenum (by mass) to a high nitrogen type 304 steel increased both rupture life and ductility; molybdenum levels of up to 0.2 % by mass had little effect on rupture life although the ductility was increased (Goodell *et al.* 1967).

The reason for the effect of molybdenum on creep properties is well documented. It not only acts as a substitutional hardener capable of being retained in the matrix for long times even at high temperature, but also strengthens the matrix by promoting the precipitation of the carbide $M_{23}C_6$ (Kawabe *et al.* 1968).

2.4. *Effect of phosphorus*

Additions of phosphorus to austenitic steels have been found to affect creep rupture properties markedly (Matsuo *et al.* 1973; Rundell & Raudebough 1961; Yukitoshi & Yoshikawa 1973). Although they do not affect room temperature or high temperature tensile properties, additions of up to 0.057 % phosphorus (by mass) increase the stress to rupture in 4000 h in the temperature range 550–650 °C (see figure 3). The increase in creep rupture strength increases as the test temperature increases and is greater at the lower stresses. However, work on a similar steel showed that phosphorus has little effect on rupture lives up to 1000 h at 750 °C although at 650 °C an increase in rupture life was observed (Yukitoshi & Yoshikawa 1973). For type 304 and 18 %Cr10 %NiTiNb steels, it has been found that there is a limit of approximately 0.25 % phosphorus (by mass) above which no further improvement in rupture life occurs (Matsuo *et al.* 1973). In contrast, for low carbon type 304 and type 316 steels, the rupture life increases continuously with phosphorus content up to 0.57 % by mass (Rundell & Raudebough 1961) and for an 18 %Cr10 %NiTiNbMo steel the rupture life increases with additions up to 0.3 % by mass (Matsuo *et al.* 1973). These levels are, however, far in excess of commercially acceptable limits.

The increase in creep strength found on adding phosphorus to the type 316 steel is, however, coupled with a decrease in ductility particularly at the lower stresses, when the strengthening effect of phosphorus is greatest. This decrease in ductility on adding phosphorus is a feature generally found in austenitic steels (Matsuo *et al.* 1973; Rundell & Raudebough 1971).

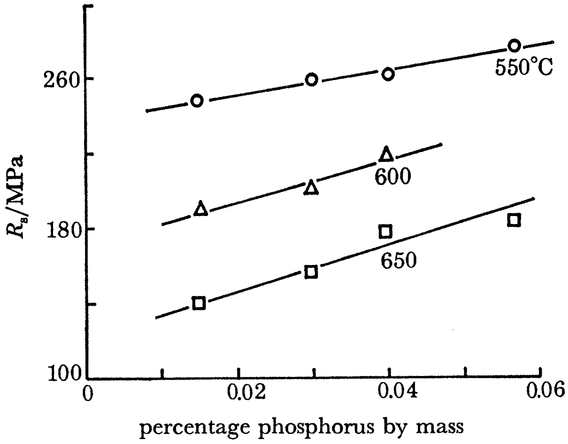

FIGURE 3. Effect of phosphorus on the stress to rupture in 4000 h, R_s, for a type 316 steel.

Phosphorus has been found to alter radically the dispersion of carbide precipitate in austenitic steels (Banerjee *et al.* 1968; Kegg *et al.* 1974; Matsuo *et al.* 1973). In a 16 %Cr10 %Ni steel, phosphorus increases the nucleation and growth rate of $M_{23}C_6$ (Kegg *et al.* 1974), although it can also promote the formation of fine matrix dot precipitate (Banerjee *et al.* 1968; Kegg *et al.* 1974). This refinement of the matrix precipitate leads to an increase in intergranular strength and hence greater creep strength and increased rupture life (Matsuo *et al.* 1973).

3. EFFECT OF RESIDUALS IN FERRITIC STEELS

Despite the recent upsurge in work into the effect of residual elements on the creep properties of ferritic steels, there has been, with the possible exception of the effects of aluminium and boron, little systematic investigation into the effect of individual elements. The effect of residuals on the creep behaviour of ferritic steels is highlighted by the creep rupture behaviour of commercial and high purity casts of two CrMoV steels. The results show that low residual levels primarily affect the fracture process, resulting in an increase in creep rupture life as well as an increase in ductility with little change in secondary creep rate (Tipler & Hopkins 1976). It was found that the high purity casts had reduced levels of intergranular cavitation damage and that the onset of cavitation was delayed to higher strains and times. Similarly in $\frac{1}{2}$ %Cr$\frac{1}{2}$ %-Mo$\frac{1}{4}$ %V steels an increase in creep crack growth rate has been correlated with an increase in residual element content (Townsend 1975).

In a 2$\frac{1}{4}$ %Cr1 %Mo steel, doping with phosphorus, antimony, arsenic and tin produced a reduction in rupture ductility but showed little evidence of affecting rupture life (Bruscato 1970). Similar results have been obtained on a low residual 2$\frac{1}{4}$ %Cr1 %Mo steel to which antimony, arsenic, silicon and phosphorus and sulphur were progressively added resulting in five casts with increasing residual element contents from which those elements affecting ductility could be identified. Increasing the antimony level of the base cast to 0.021 % by mass produced a marked reduction in ductility (see figure 4). Increasing the arsenic content to 0.063 % by mass had little further effect. These levels are, however, far above commercial limits. Additionally, increasing the silicon content to 0.32 % by mass increased the rupture ductility to the level of the low residual cast. Subsequently increasing the phosphorus level to 0.019 % by mass and the sulphur level to 0.024 % by mass did not affect the ductility. Increasing the residual element content did not affect either the rupture life or the secondary creep rate significantly.

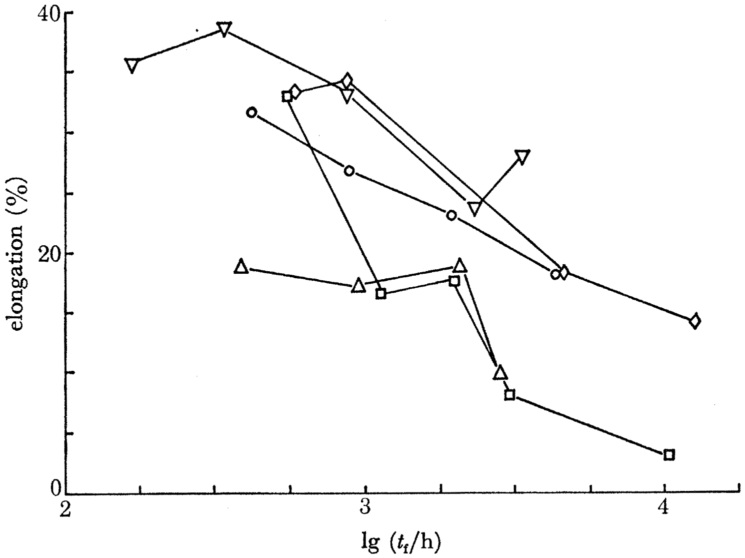

FIGURE 4. The variation of the elongation at rupture with residual content for a 2$\frac{1}{4}$%Cr1 %Mo steel at 550 °C where the base low residual cast A (○) has 0.020 % antimony (by mass) added, forming cast B (△); cast B has 0.060 % arsenic (by mass) added, forming cast C (□); cast C has 0.20 % silicon (by mass) added, forming cast D (◇); and cast D has 0.015 % phosphorus and sulphur (by mass) added, forming cast E (▽).

Reported effects of these trace elements in other low alloy ferritic steels are limited. In a 1 %Cr$\frac{1}{2}$ %V steel, antimony in the presence of manganese reduced rupture ductility with the creep strength remaining constant (Vodsedalek & Sicho 1970). Similarly, increasing the phosphorus content only slightly reduced the rupture life (Vodsedalek & Sicho 1970; Lanskaya 1976). A similar effect has been found in a 1$\frac{1}{4}$ %Cr$\frac{1}{2}$ %Mo steel when the phosphorus content was increased, although the tin and antimony content was increased simultaneously (Viswanathan 1975). Silicon additions to 1 %Cr$\frac{1}{2}$%Mo and to 0.6 %Mn steels result in a decrease in creep strength, which is thought to result from a reduction in the soluble nitrogen content (Glen & Murray 1961). However, silicon did not significantly alter the creep rate in the 2$\frac{1}{4}$ %Cr1 %Mo steel indicating that silicon affects the fracture process rather than the deformation process in this steel.

Additions of residuals to a 1 %CrMoVTiB steel, however, can have an appreciable effect not only on ductility but also rupture life. Sequential levels of the groups of residual elements copper and nickel, niobium and cobalt, and arsenic and tin were added to a base of electrolytic iron producing four casts ranging from a very pure steel to one containing all the residuals at levels typical of commercial material (see table 2). Additions of copper and nickel to the base low residual steel and arsenic and tin to the steel containing copper, nickel, niobium and cobalt decreased the stress to rupture in 2000 h (see table 2). This decrease is more pronounced at high stress than at low stress because the increased residual content increased the stress sensitivity of rupture life. Further, only the joint addition of tin and arsenic impaired rupture ductility; the other residuals had no appreciable effect. In notched specimens, increasing the residual content reduced the stress to fracture, and the effect was more pronounced than that observed with unnotched specimens when the same residuals were added (see table 2).

TABLE 2. EFFECT OF RESIDUALS ON THE RUPTURE LIFE OF A 1 %CrMoVTiB STEEL TESTED AT 550 °C

cast	residual element added	content of residual element increased (mass %)		stress to rupture in 2000 h/MPa	
		from	to	plain	notch
A	base	—	—	335.7	409.3
B	A with				
	copper	0.017	0.15	319.9	349.9
	nickel	0.01	0.17		
C	B with				
	niobium	0.004	0.011	326.6	315.5
	cobalt	0.010	0.040		
D	C with				
	arsenic	0.005	0.032	312.6	281.8
	tin	0.003	0.023		

In a $\frac{1}{2}$ %Cr$\frac{1}{2}$ %Mo$\frac{1}{4}$ %V steel tested at 600 °C, a maximum in rupture life occurred when the copper content was 0.2 % (Benes & Skvor 1972). Corresponding to the maximum there was, however, a minimum in rupture ductility. For this steel, however, increasing the copper content led to an increase in tensile strength, a feature that was not observed in the 1 %CrMoVTiB steel. Thus the explanation that the maximum in rupture life occurs at the solubility limit of copper in steel at the test temperature (Benes & Skvor 1972) would not seem appropriate. This is emphasized by results in a 1 %Cr$\frac{1}{4}$ %Mo$\frac{1}{4}$ %V steel (Lanskaya 1976) where increasing the copper content had little effect on rupture life.

The reported effect of tin on rupture properties is variable and appears to depend on the presence of other impurity elements. In a high purity 2$\frac{1}{4}$ %Cr1 %Mo steel additions of up to 0.11 % tin had no significant effect on rupture properties (Hopkins & Jenkinson 1962). In contrast, tin added to a 1 %Cr$\frac{1}{4}$ %Mo$\frac{1}{4}$ %V steel markedly reduced the rupture life and ductility (Lanskaya 1976). Similarly, in a $\frac{1}{2}$ %Cr$\frac{1}{2}$ %Mo$\frac{1}{4}$ %V steel containing copper (Benes & Skvor 1972), decreasing the tin content by 0.01 % resulted in a 10 % increase in rupture life and in a 1 %Cr$\frac{1}{2}$ %V steel (Vodsedalek & Sicho 1970) the addition of 0.024 % tin reduced both creep strength and ductility as well as increasing notch sensitivity. However, the effect of tin on creep properties in a $1\frac{1}{4}\%$Cr$\frac{1}{2}\%$Mo steel is dependent on the other residual elements present, particularly boron (Viswanathan 1975, 1977), making it difficult to identify the role of tin on creep properties in this steel.

Some explanation of the effect of residuals on creep rupture properties, particularly of phosphorus and tin has come from the use of Auger electron spectroscopy. In a $2\frac{1}{4}\%$Cr1%Mo steel containing 0.021 % phosphorus, 0.026 % tin and 0.045 % arsenic (percentages by mass) heat treated to simulate heat-affected zone structures, both phosphorus and tin have been found at prior austenite grain boundaries after creep testing at 550 °C (Needham & English 1978). The presence of tin was detected in specimens with rupture lives greater than 100 h and was coincident with a marked decrease in rupture ductility. This emphasizes the strong connection between temper embrittlement and creep embrittlement found previously (Bruscato 1970; Townsend 1975) and provides an explanation of the reduced ductility resulting from increased grain boundary cavitation associated with steels containing high levels of residual elements.

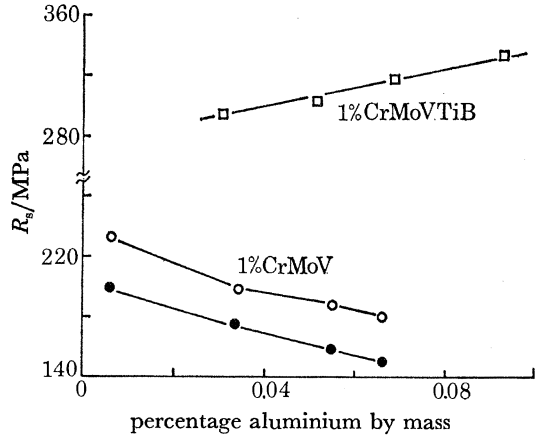

FIGURE 5. The effect of aluminium on the 2000 h rupture stress, R_s, at 550 °C for plain (o) and notched (•) specimens of a 1 %CrMoV steel and for plain (□) specimens of a 1%CrMoVTiB steel.

Variations in rupture properties in ferritic steels are not necessarily due to those elements that are commonly associated with temper embrittlement and segregation to prior austenite grain boundaries. Elements such as aluminium and boron can have a marked influence on rupture properties.

In a 1 %CrMoV steel increasing the aluminium content was found to increase the stress sensitivity of rupture life; thus although increasing the aluminium content decreases the stress to rupture in 2000 h (see figure 5), the effect of aluminium is not as great at long fracture life as at short fracture life. In the stress range 154–340 MPa these steels were notch weakened. The effect of aluminium in decreasing the creep rupture life in notch specimens is similar to that in plain specimens (see figure 5). Similar effects have been found for aluminium on rupture properties in CrMo steels (Yukitoshi & Nishida 1973) and other CrMoV steels (Lanskaya 1976; Ratliff & Brown 1967).

It is clear that, although there is evidence that aluminium increases the matrix strength of a steel as a result of a refinement of the precipitate structure (Ratliff & Brown 1967), it can have other effects such as grain refinement which can be detrimental to creep rupture life (Yukitoshi

& Nishida 1973). Further, aluminium additions can remove nitrogen from solution as has been shown in CMn and CrMo steels (Glen & Murray 1961) which will result in a loss in creep strength.

The adverse effect of aluminium is, however, not universal. In a 1%CrMoVTiB steel, increasing the aluminium content results not only in an increase in stress to rupture in 2000 h (see figure 5), but also in an increase in rupture ductility. Similar increases in creep rupture strength have been observed in a $1\frac{1}{4}$%Cr1%Mo$\frac{1}{4}$%V steel when additions of aluminium and nitrogen were made, although for this steel the rupture ductility was impaired (Viswanathan & Beck 1975). Further, a minimum in the 1 h rupture stress has been found in CrMoV steels tested at 690 °C, with the rupture stress increasing for aluminium contents above 0.035 % (Townsend 1975). Clearly the effect of aluminium and the allied effect of nitrogen is complex, particularly in precipitate strengthened steels.

Not all residual elements are, however, detrimental to creep properties. Additions of boron to a 1%CrMoV steel (Keown 1973) and to a 1%Cr1 %Mo$\frac{3}{4}$%VTi steel (Stone & Murray 1965) improved creep ductility by retarding the onset of the ductility minimum associated with precipitation of vanadium carbide. Further, for the 1%Cr1%Mo$\frac{3}{4}$%VTi steel, boron also improves the notched stress rupture properties to an extent that the material becomes notch strengthened (Stone & Murray 1965). However, more recent work indicates that in a similar steel there is an optimum titanium and boron content at which a peak in rupture life is attained.

It is thought that boron refines the precipitate morphology, thus eliminating the grain boundary precipitate free zone found in this steel (Stone & Murray 1965). This would increase the strength of the grain boundary region thus reducing the susceptibility to brittle fracture. In a 1%Cr$\frac{1}{2}$%Mo steel, however, the effect of boron on creep rupture life and ductility depends on the other impurity elements present (Viswanathan 1977), although when associated with titanium, boron will increase rupture life irrespective of the impurity elements present. These results were explained in terms of the effect of titanium and boron on the bainite hardenability and emphasizes the complexity of the effect of residual elements on structure and strength of materials.

4. Summary

The effect of residual elements on the elevated temperature properties in austenitic and low alloy ferritic creep resistant steels has been reviewed. Their effect is such that much of the scatter in creep properties for a particular grade of steel may result from variations in residual element content.

In austenitic steels, additions of boron, molybdenum, nitrogen and phosphorus are found to be beneficial to creep strength and rupture life. Nitrogen and phosphorus are, however, deleterious to rupture ductility although boron and molybdenum are not and can for certain steels be beneficial.

The effect of individual residual elements on the high temperature properties of ferritic steels appears to depend on the presence of the other impurity elements. However, in a 1%CrMoV-TiB steel, additions of copper plus nickel and arsenic plus tin reduce rupture life, their effect being greater the shorter the creep life. Only the arsenic plus tin addition affected rupture ductility, these elements having a similar effect in other steels. In a $2\frac{1}{4}$%Cr1%Mo steel antimony reduces rupture ductility but silicon increases it. Similarly, boron in the presence of titanium increases both rupture life and ductility in CrMoV steels, and aluminium, although generally detrimental to rupture life and ductility, has in a 1%CrMoVTiB steel been found to be beneficial in both.

Tin and phosphorus have both been identified at prior austenite grain boundaries as a result of creep by Auger electron spectroscopy.

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