HIGH PERFORMANCE SINGLE CRYSTAL SUPERALLOYS

DEVELOPED BY THE d-ELECTRONS CONCEPT

N.Yukawa, M.Morinaga, Y.Murata, H.Ezaki, and S.Inoue*

Toyohashi University of Technology, Toyohashi, Aichi 440, Japan

*Numazu College of Technology, Numazu, Shizuoka, 410, Japan

Abstract

New single crystal superalloys of Ni-10at%Cr-12at%Al-Ti-Ta-W-Mo-Re have been developed by the d-electrons concept, which was devised on the basis of molecular orbital calculations of Ni based alloys. The process for the design of single crystal superalloys introduced in our previous study was modified to a more simple one and applied to the present study. The alloys were designed so as to have an optimum combination of the properties such as high temperature strength and ductility, hot corrosion resistance, density and cost of the alloys, while holding good fabricability and phase stability. The d-orbital energy level (Md) of alloying elements was utilized in order to predict and control the occurence of the undesired eutectic γ' and the topologically close packed (TCP) phases in alloys. The alloying effect of Cr, Ti, Al, Re and Co on the properties of single crystal alloys was investigated systematically. Based on these results several single crystal superalloys were designed. Some of them have well balanced properties, compared to the various alloys so far developed. In addition, several important factors were discussed for strengthening single crystal superalloys.

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Introduction

A number of single crystal superalloys have been developed in several countries during the past decade (1). However, these development seems to be relied on several empirical methods. Further advances in the method of alloy design are necessary for the future improvement in the temperature capability of single crystal turbine blades and vanes (1,2).

Recently we have developed a new d-electrons concept which is based on the theoretical calculations of the electronic structure of alloys (3-5), and applied to the design of superalloys (6-8). In a previous study, a new process was proposed for the design of single crystal superalloys (8). In this process, fabricating limitations of single crystals are considered in addition to the phase stability limitations of alloys. Following this design process, several single crystal superalloys with a long creep-rupture life and high corrosion resistance were developed successfully.

In the present study, a more simplified method was devised and applied to the design of high performance single crystal superalloys. Some developed alloys exhibited an optimum balance in the properties which are required for the high temperature applications.

Experimental Procedure

Ni-Cr-Al-Ti-Ta-W-Mo-Re-Co alloys with a variety of compositions were investigated together with some reference alloys listed in Table 1.

Table 1 Chemical compositions of reference alloys.

Alloy	Composition, wt% Ni	Cr	Al	Ti	Ta	W	Mo	Re	Co	Country
PWA 1480	bal.	10.0	5.0	1.5	12.0	4.0	-	-	5.0	U.S.A.
NASAIR 100	bal.	9.0	5.8	1.2	3.3	10.5	1.0	-	-	U.S.A.
CMSX-2	bal.	8.0	5.6	1.0	6.0	8.0	0.6	-	4.6	U.S.A.
CMSX-4	bal.	6.6	5.6	1.0	6.5	6.4	0.6	3.0	9.6	U.S.A.
MXON	bal.	8.0	6.1	-	6.0	8.0	2.0	-	5.0	France
TMS-1	bal.	5.5	5.2	-	5.1	16.6	-	-	7.5	Japan
TMS-12·1	bal.	6.6	4.6	-	8.1	13.5	-	-	-	Japan
SC-53A	bal.	6.9	5.5	-	5.9	5.9	5.1	-	-	Japan

Both poly- and single-crystal specimens of these alloys were prepared. Poly-crystal specimens were prepared by a tri-arc furnace. Single crystal specimens were grown by the withdrawn process at a growth rate of 10cm/h and a temperature gradient of about 120°C/cm. The single crystal bars of 11mm in diameter was obtained, and its axial direction was near the [001] direction; the deviation from this direction was less than 10° for all the crystals used for the present investigation.

A series of experiments was carried out with these specimens after heat-treatments, 1300°C/4hrs, B.A.C. + 1050°C/16hrs + 850°C/48hrs, A.C.; here, B.A.C. means the blast air cooling.

1) Solidus and solvus temperatures and melting range of alloys, ΔT, were measured using a differential thermal analysis (DTA). The magnitude of heat-treatment window (H.T.W.) that is defined by the difference, $(T_{solidus} - T_{\gamma' solvus})$, were then determined.

2) Volume fraction of the γ' phase and the partitioning ratios of alloying elements between the γ and γ' phases were measured using an EPMA. This analysis was carried out with the residues extracted from as-grown specimens.

The extraction was performed using a potentiostatic apparatus under the condition of a constant potential in an 1% ammonium sulfate - 1% tartaric acid aqueous solution.
3) Hot corrosion resistance was examined using single crystal specimens (5× 10×1mm), surface-coated with an Na_2SO_4 - 25%NaCl salt by the amount of 20mg/cm^2. The thermogravimetric measurment was performed at 900°C in the flow of air (50cc/min). The hot corrosion index (H.C.I.) was then defined as the weight gain of the specimen (mg/cm^2) after 20 hrs exposures, and used as a measure of the hot corrosion resistance. The corrosion resistance of alloys increases with decreasing this index.
4) Creep-rupture strength was tested at 1040°C under a constant load of 14kgf/cm^2.
5) Tensile strength was also examined at 982°C.
6) Precipitation behaviors of the γ', eutectic γ', α-W and topologically close packed (TCP; μ or σ) phases were investigated. In order to examine phase stability with respect to the α-W and TCP phases in the γ phase, both poly- and single-crystal specimens were aged for 500hrs at 982°C after the solution-treatment at 1300°C.
7) Densities of alloys were computed according to the Hull's regression equation (9).
8) Cost of alloys was calculated using the following equation:

$$\begin{aligned}\text{Cost of alloy} = {} & (0.7(\text{wt\%Ni}) + 1.3(\text{wt\%Cr}) + 0.35(\text{wt\%Al}) \\ & + 2.0(\text{wt\%Ti}) + 35.0(\text{wt\%Ta}) + 6.0(\text{wt\%W}) \\ & + 0.5(\text{wt\%Mo}) + 100.0(\text{wt\%Re}) + 3.0(\text{wt\%Co}))/100, \text{yen/g}, \end{aligned} \quad (1)$$

where the coefficient in this equation indicates the price of each metal (yen/g) in June 1987 in Japan. The price might be changed by many factors, but for comparison it was fixed in this study.

Alloy Design

In the alloy design devised in our previous study (8), two types of limitations were considered. The one is a limitation for the fabrication, and the another is a limitation for the phase stability which can be treated by the d-electrons concept. Following this design process, the optimum compositional field was obtained for the single crystal superalloys of Ni-10at%Cr-12at%Al-1.5at%Ti-Ta-W-Mo. The result is represented in the coordinates of Ta and W+(Mo) content, as shown Fig.1.

In the figure, the fabricating limitation was shown by the two lines, $\Delta T \leq 50$°C and H.T.W. ≥ 20°C. The former limitation is necessary to grow single crystal superalloys, and the latter limitation is required to permit a solution-treatment process.

The phase stability limitation was shown by three lines. Two of them were expressed by the average d-orbital level of alloying transition metal, $\overline{Md}$, and that is defined as,

$$\overline{Md} = \Sigma X_i \, (Md)_i. \quad (2)$$

Here, X_i and $(Md)_i$ are the atomic fraction and Md value of i element, respectively. Table 2 shows Md values together with the values of the another parameter, bond order between M and Ni (called Bo), which will be discussed later.

The microstructural observation was performed for the alloys which were solidified in a controlled way through the single crystal growth or the constant cooling (5°C/min) in the DTA experiment. It was found that the

eutectic γ′ phase disappeared by the solution-treatment at 1300°C when its volume fraction was less than 2%. (In the case of the arc-melted specimens, the boundary increased to 4%.) This condition could be expressed using an inequality relationship, $\overline{Md}t \leq 0.985$, where $\overline{Md}t$ is the $\overline{Md}$ value calculated from the total alloy composition.

Table 2 List of Md and Bo values.

	Element	Md	Bo		Element	Md	Bo
	Ti	2.271	1.098		Zr	2.944	1.479
	V	1.543	1.141	4d	Nb	2.117	1.594
	Cr	1.142	1.278		Mo	1.550	1.611
	Mn	0.957	1.001		Hf	3.020	1.518
3d	Fe	0.858	0.857	5d	Ta	2.224	1.670
	Co	0.777	0.697		W	1.655	1.730
	Ni	0.717	0.514		Re	1.267	1.692
	Cu	0.615	0.272	Others	Al	1.900	0.533
					Si	1.900	0.589

The another $\overline{Md}\gamma$ was calculated from the composition of the residual γ matrix following the same process described in the previous study (8). Microstructural observation of alloys aged for 500hrs at 982°C revealed that the TCP phase (in this case, μ phase) could be suppressed when $\overline{Md}\gamma \leq 0.93$, and hence this is the another limitation for phase stability.

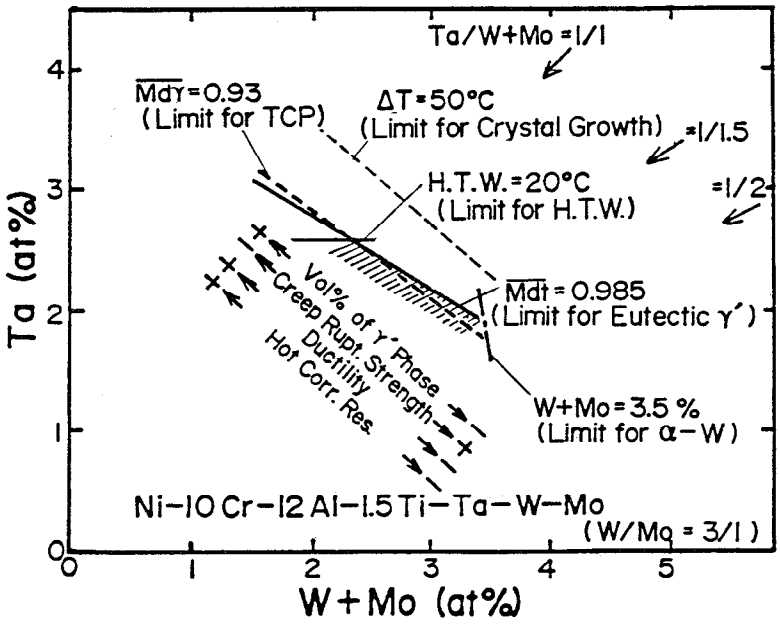

Fig.1 Phase stability diagram of single crystal superalloys.

In the previous study, the condition, $\overline{Md}\gamma(Ta+W) \leq 0.105$, was used as the limitation for the precipitation of the α-W phase. In the subsequent study, however, it was found that W+Mo ≤ 3.5at% was the better limitation for Ni-10at%Cr-12at%Al-Ti-Ta-W-Mo alloys, then it was used in this study.

As can be seen in Fig.1, both lines, $\overline{Md}t=0.985$ and $\overline{Md}\gamma=0.93$, were overlapped, and may be considered practically as one line. Therefore, only the $\overline{Md}t$ was employed in the present alloy design in order to represent the phase stability. This criterion seems to be applicable for the alloys containing 10∿13at% Cr. When the Cr content decreases to 9∿7at%, the $\overline{Md}t$ increases to 0.986, as is seen in CMSX-4.

In the figure, trend of the changes in alloy properties with Ta and W+Mo

contents is also illustrated. The vol% of the γ' phase increased with Ta content, and its maximum value was 67vol%. Creep-rupture strength increased with W+Mo content. By contrary, ductility decreased with W+Mo content. In addition, hot corrosion resistance increased with Ta content. From the above trend, it was concluded that these properties could be controlled by the Ta/W+Mo ratio. (This ratio was calculated in at%.) In the present study, alloys were designed in the range of Ta/W+Mo atomic ratio from 1/1 to 1/2. The optimum compositional field of the alloys is indicated by the shaded area in Fig.1.

The process of alloy design employed in this study is shown in Fig.2. W/Mo atomic ratio was kept at 3/1 in the calculation. Density and cost of alloys were also calculated together with the γ' vol%, the compositions of the both γ and γ' phases, and the $\overline{Md}\gamma$ value.

For the development of single crystsl superalloys, the following five major properties were considered: 1) creep-rupture life, 2) creep-rupture elongation, 3) H.C.I., 4) density, 5) materials cost. An optimum combination of these properties was searched for in the present design.

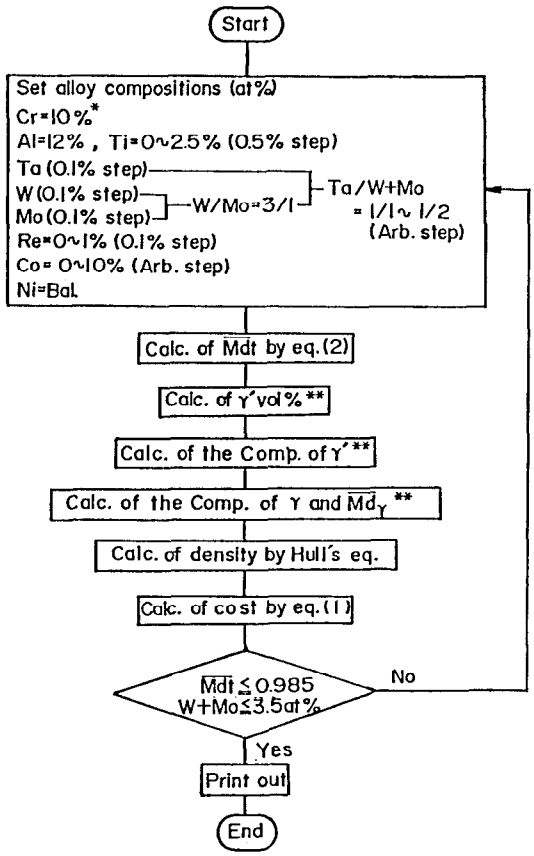

Fig.2 Flow chart for the design of single crystal superalloys.
* In this study, Cr content was changed up to 16%.
**For the calculation procedure, see Ref.(8).

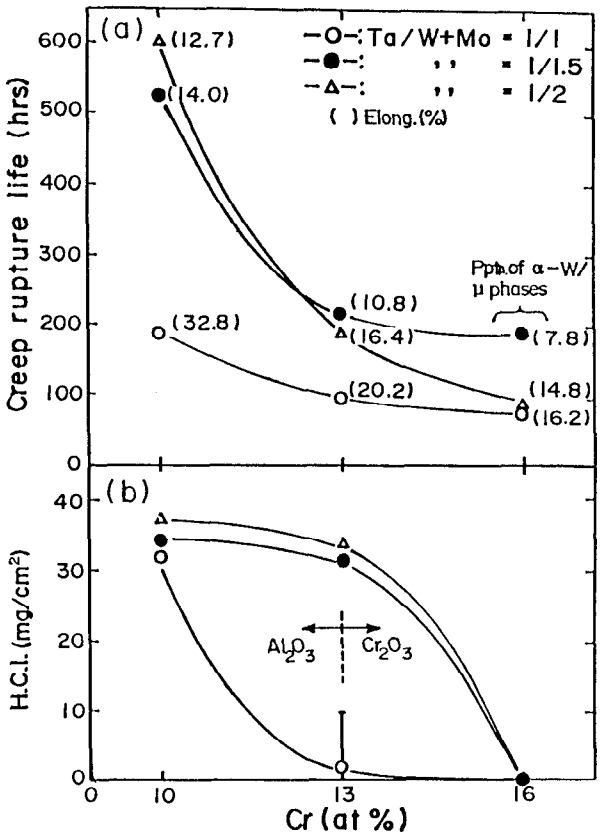

Fig.3 Effect of Cr on (a) the creep-rupture strength and (b) the hot corrosion resistance of Ni-10~16Cr-12Al-1.2Ti-1.2~2.7Ta-1.2~2.9W-0.4~1.0Mo (at%) alloys.

Alloying Effects on the Properties of Single Crystal Superalloys

The alloying effects of elements on the properties of single crystal superalloys were investigated for the designed alloys and some reference alloys. Here, only the relevant results are outlined. The detailed explanation on the hot corrosion resistance will be given elsewhere (10).

Effect of Cr and Ta/W+Mo atomic ratio

Fig.3 shows the effect of Cr content on the creep-rupture properties and the hot corrosion resistance of Ni-10∿16Cr-12Al-1.2Ti-1.2∿2.7Ta-1.2∿2.9W-0.4∿1.0Mo (at%) alloys. These experimental alloys had different Ta/W+Mo atomic ratios of 1/1, 1/1.5, and 1/2.

As shown in Fig.3 (a), creep-rupture life decreased with Cr content and with Ta/W+Mo atomic ratio except for the alloys with higher Cr content. Rupture elongation indicated by the number in parenthesis decreased with Cr content except for some alloys with the Ta/W+Mo atomic ratio of 1/2, but increased with Ta/W+Mo atomic ratio. As is shown in the figure, the precipitation of the α-W and μ phases was observed in the 16at%Cr alloys. Relatively small elongation of these alloys may be attributed to the precipitation of these phases. Fig.3 (b) shows the change of H.C.I. with Cr content. The H.C.I. decreased with Cr content, and became zero at 16at%Cr. The H.C.I. also decreased with Ta/W+Mo atomic ratio. Its decrease was remarkable for the alloys with the Ta/W+Mo atomic ratio of 1/1. Data was scattered at 13at%Cr, as shown by an error bar. This may be due to the occurence of the transition of the oxide formation from Al_2O_3-type to Cr_2O_3-type at this Cr content (11), and the complex oxidation mechanism was supposed to be operated at this composition.

Effect of Ti and Al

The effect of Ti content on the H.C.I. is shown in Fig.4 for Ni-10∿12Cr-11.5∿13Al-0∿2.5Ti-1.2∿4.0Ta-1.3∿3.5W-0∿0.6Mo-0∿5.2Co (at%) alloys. The H.C.I. showed a minimum at about 1.5at%Ti. Therefore, the Ti content was varied in the range of 1.2∿1.5 at% in this design.

Within the alloys investigated, the H.C.I. changed scarcely with Al. Strength properties decreased with increasing Ti and Al contents since the solubility limit of Ta, W and Mo in the γ phase decreased.

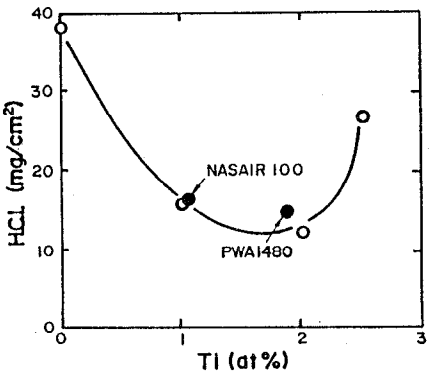

Fig.4 Effect of Ti on hot corrosion resistance of Ni-10∿12Cr-11.5∿13Al-0∿2.5Ti-1.2∿4.0Ta-1.3∿3.5W-0∿0.6Mo-0∿5.2Co (at%) alloys.

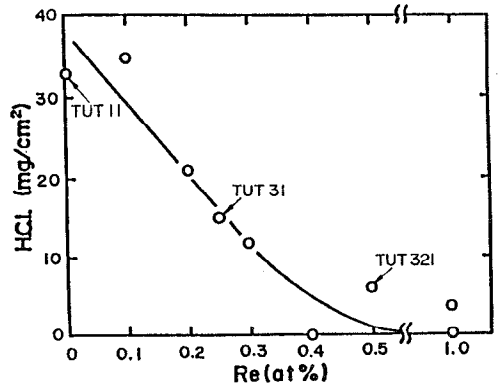

Fig.5 Effect of Re on hot corrosion resistance of Ni-9∿12Cr-11∿12Al-1.2∿2.5Ti-1.1∿2.7Ta-1.9∿3.8W-0.64∿0.67Mo-0.1∿1.0Re (at%) alloys.

Effect of Re

Fig.5 shows the effect of the Re addition on the H.C.I. of Ni-9∿12Cr-11∿12Al-1.2∿2.5Ti-1.1∿2.7Ta-1.9∿3.8W-0.64∿0.67Mo-0∿1.0Re (at%) alloys. The addition of Re to the alloys up to 1at% reduced the H.C.I. remarkably. However, the Re addition more than 0.25at% into the alloys containing 10at%Cr caused the phase instability. The eutectic γ′ phase never disappeared after solution-treatments, and also the excess precipitation of the α-W phase took place. As the result, rupture life and elongation

decreased greatly. Thus, the maximum amount of Re was considerd to be 0.25at%.

Effect of Co

The effect of Co on the single crystal superalloys was investigated by substituting Co for Ni in TUT 92 and CMSX-4. The substitution was made so that the alloys have the same $\overline{Md}t$ value or the same total W+Mo content as TUT 92. The results are summarized in Table 3. The chemical compositions, W+Mo content, Ta/W+Mo atomic ratio, $\overline{Md}t$, cost , density, creep-rupture properties and the H.C.I. were listed in the table.

Table 3 Effect of Co on the properties of TUT 92 and CMSX-4.

Alloy	Composition, wt%								
	Ni	Cr	Al	Ti	Ta	W	Mo	Re	Co
TUT 92	bal.	8.7	5.4	1.2	6.2	7.1	1.2	0.8	-
TUT 921	bal.	8.7	5.4	1.2	6.2	7.1	1.2	0.8	8.8
TUT 922	bal.	8.7	5.4	1.2	5.7	6.5	1.1	0.8	8.9
CMSX-4	bal.	6.6	5.6	1.0	6.5	6.4	0.6	3.0	9.6
CMSX-4 (0Co)	bal.	6.5	5.6	1.0	7.1	7.0	0.7	3.0	-

Alloy	W+Mo at%	Ta/W+Mo AT.ratio	$\overline{Md}t$	Dens. g/cm^3	Cost yen/g	Creep-Rup. life(h)	Creep-Rup. El.(%)	H.C.I. mg/cm^2
TUT92	3.1	1/1.5	0.985	8.62	4.1	950	10.0	13.3
TUT921	3.1	1/1.5	0.990	8.60	4.3	680	8.7	6.0
TUT922	2.8	1/1.5	0.985	8.54	4.1	570	17.0	3.9
CMSX-4	2.5	1/1.1	0.986	8.78	6.5	610	19.5	0.0
CMSX-4 (0Co)	2.8	1/1.1	0.986	8.86	6.5	950	17.3	15.2

For TUT 921, containing 8.8wt%(9at%)Co but the same W+Mo content (3.1at%) as TUT 92, a considerable decrease was observed in the rupture life, the rupture elongation and H.C.I. index. This was interpreted as due to the eutectic γ' phase which was retained even after heat-treatments probably owing to the higher $\overline{Md}t$ value (0.990) than the critical value of 0.985 (see Fig.1).

Also, as is seen in TUT 922, when the Co addition was made under the same $\overline{Md}t$ value, rupture life and the H.C.I. further decreased, whereas the rupture elongation increased. By contrary, the elimination of 9.6wt%(9.9at%)Co from CMSX-4, resulted in the increase of rupture life and H.C.I., but caused the slight decrease of rupture elongation.

From these results it is concluded that the addition of Co decreases the solubility limit of Ta, W and Mo in the alloy, which led to the decrease of the strength properties. On the other hand, hot corrosion resistance of the alloy increased to some extent with the addition of Co.

The Properties of Developed Single Crystal Superalloys

The compositions and the properties of some developed alloys are shown in Table 4. All of these alloys consisted of homogeneous microstructure after the heat-treatment. Except for TUT 321, no precipitation of the μ and α-W phases was observed after the aging for 500hrs at 982 °C or after the creep-rupture test.

Creep-rupture life increases with increasing W+Mo content and with decreasing Ta/W+Mo atomic ratio. The addition of Re drastically increases creep-rupture life and hot corrosin resistance, but this also causes the increase of density and materials cost.

Table 4 Chemical compositions and the properties of developed alloys.

Alloy	Composition, wt% Ni	Cr	Al	Ti	Ta	W	Mo	Re	W+Mo at%	Ta/W+Mo AT.ratio	Dens. g/cm^3	Cost yen/g	Creep-Rup. life hrs	Creep-Rup. El. %	H.C.I. mg/cm^2
TUT 11	bal.	8.6	5.4	1.0	8.1	6.2	1.1	-	2.7	1/1	8.60	3.9	282	15.9	33.5
TUT 31	bal.	8.6	5.4	1.0	7.9	6.0	1.0	0.8	2.6	1/1	8.65	4.6	1700	7.0	15.4
TUT 321*	bal.	8.6	5.4	1.0	7.7	5.9	1.0	1.5	2.6	1/1	8.71	5.3	674	14.4	6.4
TUT 22	bal.	8.7	5.4	1.2	7.5	5.7	1.0	0.2	2.5	1/1	8.56	3.8	219	16.6	22.7
TUT 52	bal.	8.7	5.4	1.2	6.9	6.5	1.1	0.2	2.7	1/1.25	8.57	3.7	574	9.9	27.5
TUT 82	bal.	8.7	5.4	1.2	6.3	7.2	1.3	0.2	3.1	1/1.5	8.58	3.5	652	7.4	36.4
TUT 92	bal.	8.7	5.4	1.2	6.2	7.1	1.2	0.8	3.1	1/1.5	8.62	4.1	950	10.0	13.3

* Precipitation of the μ and α-W phases was found after the aging and creep-rupture test.

In Fig.6, five properties of the developed alloys, TUT 31 and 92, are illustrated using a pentagonal axis, and compared with the data of typical reference alloys. It is apparent that TUT 92 exhibited the more balanced properties than the reference alloys. As for the reference alloys, it is found that creep-rupture life increases with W+Mo content. Poor hot corrosion resistance of MXON (12) and TMS (13,14) alloys may be attributed partially to lack of Ti. As for TMS alloys, low Cr and high W+Mo contents, and low values of Ta/W+Mo atomic ratio probably cause the very poor hot corrosion resistance though they exhibited excellent strength properties. The addition of 3wt%Re in CMSX-4 results in excellent hot corrosion resistance in spite of the reduced Cr content of 6.6wt%. However, it is penalized by the increase of cost and density.

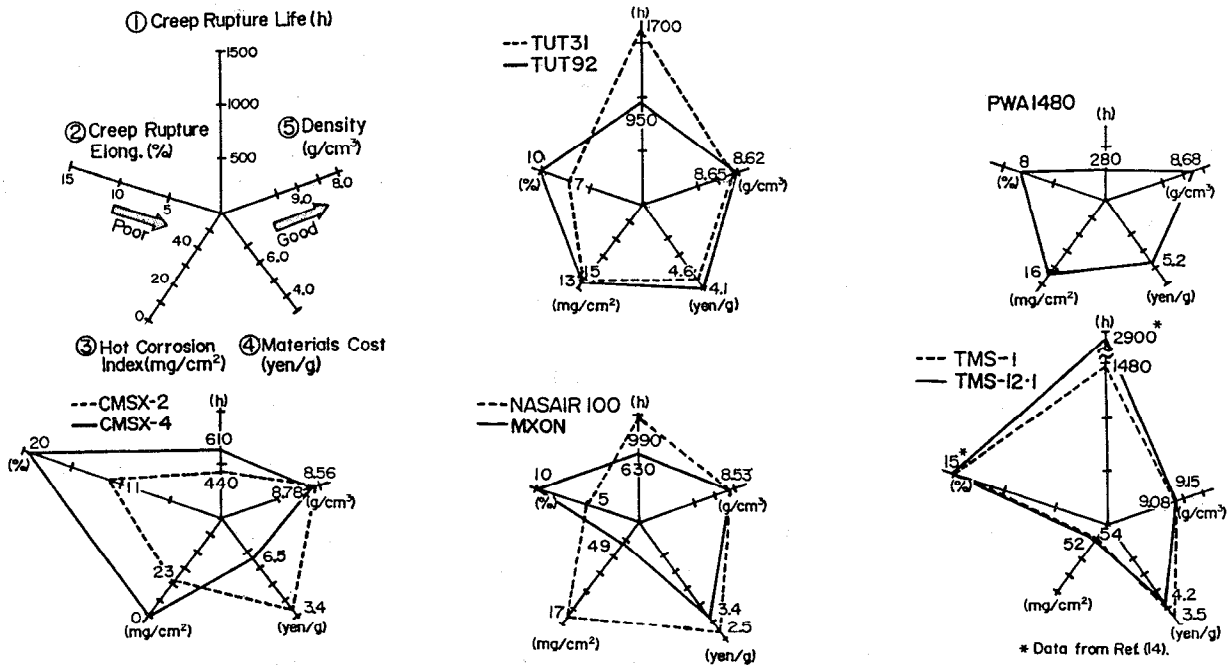

Fig.6 Comparison of the five properties of typical developed alloys with those of the reference alloys.

Discussion

It was demonstrated that a new method utilized the $\overline{Md}t$ is a nice tool for the design of single crystal superalloys. In Fig.7 measured solvus temperatures are plotted as a function of $\overline{Md}t$ for Ni-10/11Cr-12/14Al-0/2Ti-1∿4Ta-1.25∿3.50W-0.81∿0.88Mo-0∿7.5Co (at%) alloys and some of reference

alloys. The vol% of the eutectic γ' phase in alloys solidified at a constant cooling rate of 5°C/min is also shown in the figure. The solvus temperature increases linearly with $\overline{Mdt}$ up to 0.985 and appears to stay constant for higher $\overline{Mdt}$. Vol% of the eutectic γ' increases with $\overline{Mdt}$. As stated before, to devoid of this phase after solution-treatments the critical amount should be less than 2% and this condition is achieved around the $\overline{Mdt}$ value of 0.985. So, this criterion for $\overline{Mdt}$ was employed in the present design.

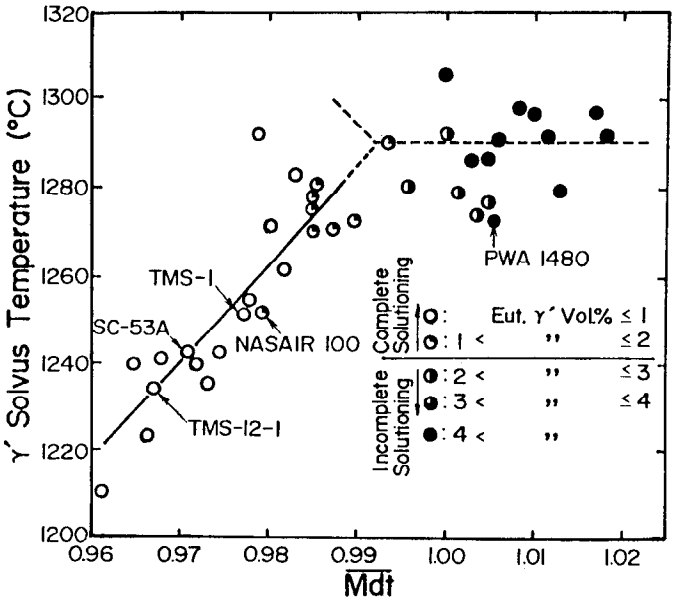

Fig.7 Relation between volume % of the eutectic γ' phase and γ' solvus-temperature of Ni-10/11Cr-12/14Al-0/2Ti-1∿4Ta-1.25∿3.50W-0.81∿0.88Mo-0∿7.5Co (at%) alloys and the reference alloys.

Many guidlines based on the concepts of physical metallurgy have been proposed for the design of high strength superalloys. For example, the lattice misfit between the γ and γ' phases was often taken into account in the alloy design (13). As described in Fig.1, however, the trend of the change in alloy properties with Ta and W+Mo contents implied that the solution strengthening of the γ phase is the most crucial factor for strengthening single crystal superalloys. The effect of the γ/γ' lattice misfit on the strengthening, of course, can not be neglected, but this appears to be subsidiary compared to the strengthening effect of the γ phase through the solutioning of refractory metals such as W, Mo and Re. This effect may be related to the magnitude of bond order (Bo). As shown in Table 2, these elements have high covalent bond-strength with Ni, and they are preferably partitioned to the γ phase and effective in strengthening it. The elimination of Co resulted in the decrease of $\overline{Mdt}$, which allows the further solutioning of W, Mo and Re into the matrix and strengthens the alloy. This is in good agreement with the experimental results for MXON alloy (12). Utilizing this concept, it is possible to tailor single crystal alloy compositions for specific applications.

Conclusions

Based on an electronic theory, a new design process for single crystal supperalloys was devised and applied to the development of high performance single crystal alloys. It was concluded that the d-electrons concept makes the alloy design more efficient and accurate compared to the currently used methods standing on several empirical rules and many trial-and-error experiments.

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