actual mechanism of flaw formation. The possibility that these results could be explained in terms of thermal shock (i.e. the production of internal stresses due to a temperature gradient within the fibre) cannot be dismissed, although the following factors cast some doubt on this possibility: (a) the fibres have a small diameter (~8 μm) and good thermal conductivity; (b) although little thermal shock can have occurred in the case of the fibre cooled from 2000°C at 2°C min^{-1}, its strength is reduced compared to the original fibre, indicating the likelihood of another mechanism for flaw formation; (c) recent work by Bullock [9] showed flaw formation to be associated with rapid temperature change, but his results give clear evidence [10, 11] that thermal shock effects did not cause the flaws. The results were, however, explicable in terms of the crystallite interaction model of Jones and Duncan [1-3].

We can be certain then, that the voids observed by electron microscopy [4] do not represent the sole factor governing fibre strength. It seems likely, however, that the flaws generated during cooling will often be associated with existing voids and, for this reason, the voids probably represent an important structural factor in relation to the influence of cooling rate on fibre strength.

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Received 21 August
and accepted 1 September 1973

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On a statistically-designed nickel-base alloy

In a paper a few years ago, Collins *et al* [1] described the development of the alloy NASA TRW VI A through the application of statistically-designed experiments. By having recourse to such experiments they were able to screen the effects of fourteen elements in about a 2 year period, and so produce an alloy which is amongst the strongest nickel-base superalloys presently available [2, 3]. It is strengthened by two MC-type and one $M_{23}C_6$-type carbide together with γ' [4].

The experiments which were used fell into five series and comprised two types: the preliminary studies (series I-III in their notation which we retain) were based on Latin Square designs and were followed by a detailed optimization (series IV and V) using the fractional factorial approach. The results of the latter were then incorporated into a regression analysis for the four most effective elements, namely Cr, W, Ta and Hf. The stationary point of the resulting equation was labelled alloy VI A, tested experimentally, and found to be better than all development alloys.

As part of a study on the use of statistical design in alloy development, we have re-examined the design and analysis of these experiments of Collins *et al* [1]. The object of the present note is to point out that the analysis of their Latin Square designs is incorrect and to remark on the consequences of a corrected analysis. It should be pointed out at this stage that the final alloy VI A is not affected by the revision.

In their study, Collins *et al* [1] cast-up each alloy melt as a tensile bar cluster. These preforms were then ground down into test samples, some of which were tested as follows:

(a) SRL: stress rupture life (three samples) at

TABLE I SRL of series I containing 4.5% Ti and 1.0% Ti

	Sum of squares	Degrees of freedom	Mean square	*F*-ratio
Original analysis – Table A 1 in [5]				
Mo	1.9682	2	0.9481	28.52‡
W	5.3706	2	2.6853	77.83‡
Ta	16.0258	2	8.0129	232.26‡
Residual	0.6907	20	0.0345	
Total	24.0553	26		
Present analysis				
Mo	1.9682	2	0.9481	3.21
W	5.3706	2	2.6853	8.77
Ta	16.0258	2	8.0129	26.19*
Residual between melts	0.6119	2	0.3060	69.55†
Between bars within melts	0.0788	18		
Total	24.0553	26		

Significant at *95%, †99.9%.

TABLE II (all compositions in wt %)

	Collins *et al*		Present work	
	SRL	TE	SRL	TE
Series I				
4.5 Al 1.0 Ti levels				
Mo (1.0, 4.5, 8.0)	pos.	—	—	—
W (1.0, 5.5, 10.0)	pos.	—	—	—
Ta (1.0, 4.5, 8.0)	pos.	—	pos.	—
6.3 Al 1.0 Ti				
Mo	—	neg.	—	—
W	—	neg.	—	—
Ta	pos.	max. at 4.5	—	—
6.3 Al 1.6 Ti				
Mo	—	neg.	—	—
W	—	neg.	—	—
Ta	—	max. at 4.5	—	—
Al	not analysed		—	neg.
Ti	not analysed		—	—
Series II				
Nb (0, 1.0, 2.0)	—	—	—	—
Hf (0, 1.0, 2.0)	pos.	pos.	—	—
V (0, 1.0, 2.0)	neg.	max. at 1%	—	—
Series III				
Re (0, 2.0, 4.0)	—	neg.	—	—
Ru (0, 1.0, 2.0)	neg.	—	—	—
Al (4.5, 5.4, 6.3)	max. at 5.4	neg.	cut-off after 5.4	neg.

2000°F and 15000 psi (1093°C and 10.55 kg mm^{-2});

(b) TE: tensile elongation (two samples) at 1400°F (760°C). The results of these tests were used for the analysis in which it was assumed that the variation in properties within a single melt was the same or greater than between melts of the same alloy.

The Latin Square designs were used in series I-III to test the effect of three alloying elements

varied simultaneously at three levels (compositions). In series I, the added elements were Mo, W and Ta. The design was repeated in two other Squares in which the levels of Ti and Al were altered. Series II and III studied Nb, Hf, V and Re, Ru, Al respectively. In analysing the data, the assumption was made that the residual error term contained 20 degrees of freedom (Table I) which had the consequence of making the analysis oversensitive in estimating statistical significance, even though a significance level of 99% was employed in place of a more usual 95%.

A correct analysis, presented now, divides the residual error term into two components, one giving the residual variance between melts and the other representing the error between bars within melts. The decomposition of the degrees of freedom for the Analysis of Variance is:

	Degrees of freedom
Mo	2
W	2
Ta	2
Residual between melts	2
Total between melts	8
Between bars within melts	18
Total between bars	26

In most cases the residual variance between melts proved to be significantly greater than the variance between bars within melts. The effects of the three main factors were, therefore, tested using the error term of the Latin Square with its 2 degrees of freedom. We have re-analysed the original data [5] following this assignation. An example of the present analysis is compared with the original [5] in Table I.

In addition to the analysis given in the original work, it is also possible to test the effect of Al and Ti from the three Latin Squares comprising the series I alloys. The result of this analysis, together with the re-analysed Latin Squares comprising series I, II and III, is presented in Table II. Considering series I it is clear that, irrespective of the fact that the significance level is now set at the lower value of 95%, the interpretation is very different. Only two results are significant: Ta has a positive effect on the SRL (in the composition range studied) whilst Al has a negative effect on the TE. Further, the observation that the effect of Ta depends upon the levels of Ti and Al shows, moreover, that interaction effects between the elements cannot be neglected.

In series II and III the only significant element is (again) Al, where the composition range studied is the same as within series I. The present analysis gives the same result as above: there is a detrimental effect on TE. The data from the mid-point composition allowed us to make a further comparison on the SRL data between the three levels. The difference between the first pair (4.5 and 5.4 wt %) is not significant, whereas that between the second pair (5.4 and 6.3 wt %) is significantly detrimental. There seems to be a cut-off effect between this second pair (possibly a phase boundary) on the aluminium-poor side of which the element is not significant but beyond which it is deleterious. The conclusion is contrasted with the conclusion given previously [1] of a maximum beneficial effect at 5.4 wt %.

In retrospect, the use of Latin Squares in this manner does not appear to have been very successful in determining the next step in development. It would probably have been better to use factorial designs.

The later series IV-V did indeed use fractional factorial designs to estimate the effect of the individual elements and their interactions. The design used for series IV was derived from that given by Natrella [6] but was modified by selecting the alternative half-replicate during the construction. This had the advantage that only six of the eight elements involved would be simultaneously at their higher level at any one time.

In studying the data we have used a 95% significance level in place of the 99% level employed previously [1]. In practice, this has very little effect on the interpretation as seen in Table III. Specifically, the effect of carbon (which would be expected intuitively) and some interaction terms become significant in series IV and the effect of tungsten and one interaction pair term become significant in the TE of series V. However, as remarked above, these changes do not affect the series VI alloys.

It is noteworthy that Re was never shown to have a statistically significant effect. It appears in the interaction terms but, as these are confounded, no clear implications can be drawn. However, Collins [7] has since demonstrated that this element does have a beneficial effect on the alloy.

In conclusion, the use of Latin Square designs in the manner adopted by Collins *et al* [1] has

TABLE III

	Collins *et al*		Present work	
	SRL	TE	SRL	TE
Series IV				
Cr	neg.	neg.	neg.	neg.
Ta	neg.	neg.	neg.	neg.
W	neg.	—	neg.	neg.
Hf	neg.	—	neg.	—
Co	neg.	—	neg.	—
C	—	—	pos.	neg.
Zr	—	—	—	—
Re	—	—	—	—
CoHf + WRe − CrC − TaZr	neg.	—	neg.	—
CoCr + TaW − HfC − ReZr	neg.	—	neg.	—
CrTa + CoW + HfRe + CZr	neg.	—	neg.	neg.
TaHf − CoZr + CrRe − WC	—	neg.	neg.	neg.
CoTa + CrW − ReC − HfZr	—	—	—	pos.
CrHf − CoC + TaRe − WZr	—	—	—	neg.
CoRe + HfW − TaC − CrZr	—	—	—	—
	99% significance		95% significance	
Series V				
Cr	pos.	pos.	pos.	pos.
W	pos.	—	pos.	nearly pos.
Ta	pos.	—	pos.	—
Hf	—	—	—	—
Cr (−W) + Ta (−Hf)	—	—	—	pos.
Cr (Ta) + (−W) (−Hf)	—	—	—	—
(−W) Ta + Cr (−Hf)	—	—	—	—

little to recommend it. An analysis of the large number of alloys examined by them, was not successful in demonstrating the significance of the added elements. The only exceptions are Ta in only one case and Al whenever it was varied. In contrast, the fractional factorial designs permitted the development of an alloy with very good properties (NASA TRW VI A) with a minimal amount of experimental work.

The authors wish to express their gratitude to Dr R. L. Dreshfield and Dr H. E. Collins for readily making available the original data on which the present analysis was based, together with details of their more recent work.

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Received 21 August
and accepted 23 August 1973

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