

General Disclaimer

One or more of the Following Statements may affect this Document

- This document has been reproduced from the best copy furnished by the organizational source. It is being released in the interest of making available as much information as possible.
- This document may contain data, which exceeds the sheet parameters. It was furnished in this condition by the organizational source and is the best copy available.
- This document may contain tone-on-tone or color graphs, charts and/or pictures, which have been reproduced in black and white.
- This document is paginated as submitted by the original source.
- Portions of this document are not fully legible due to the historical nature of some of the material. However, it is the best reproduction available from the original submission.

Orientation and Temperature Dependence of Some Mechanical Properties of the Single-Crystal Nickel-Base Superalloy René N4 III—Tension-Compression Anisotropy

R.V. Miner, T.P. Gaab, and J. Gayda
*Lewis Research Center
Cleveland, Ohio*

and

K.J. Hemker
*University of Cincinnati
Cincinnati, Ohio*



Prepared for the
One hundred fourteenth Annual Meeting of the
American Institute of Mining, Metallurgical and Petroleum Engineers
New York, New York, February 24-28, 1985



ORIENTATION AND TEMPERATURE DEPENDENCE OF SOME MECHANICAL PROPERTIES
OF THE SINGLE-CRYSTAL NICKEL-BASE SUPERALLOY RENÉ N4
III - TENSION-COMPRESSION ANISOTROPY

R.V. Miner, T.P. Gabb, and J. Gayda
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

and

K.J. Hemker
University of Cincinnati
Dept. of Metallurgy
Cincinnati, Ohio

ABSTRACT

Single crystal superalloy specimens with various crystallographic directions along their axes were tested in compression at room temperature, 650, 760, 870, and 980°C. These results are compared with the tensile behavior studied previously. The alloy, René N4, was developed for gas turbine engine blades and has the nominal composition 3.7 Al, 4.2 Ti, 4 Ta, 0.5 Nb, 6 W, 1.5 Mo 9 Cr, 7.5 Co, balance Ni, in weight percent. Slip trace analysis showed that primary cube slip had occurred even at room temperature for the $[\bar{1}11]$ specimens. With increasing test temperature more orientations exhibited primary cube slip, until at 870°C only the [001] and [011] specimens exhibited normal octahedral slip. The yield strength for octahedral slip was numerically analysed using a model proposed by Lall, Chin, and Pope to explain deviations from Schmid's Law in the yielding behavior of a single phase γ' alloy, $Ni_3(Al, Nb)$. The Schmid's Law deviations in René N4 were found to be largely due to a tension-compression anisotropy. This is one of the sources of the Schmid's Law violations observed in $Ni_3(Al, Nb)$ which are rationalized by the model. A second effect, which increases strength for orientations away from [001], was found to be small in René N4. Analysis of recently published data on the single crystal superalloy PWA 1480 yielded the same result.

INTRODUCTION

The alloy investigated in this work was developed by the General Electric Company and is designated René N4. The nominal composition of the alloy is presented in Table I. The alloy was specifically developed for application as cast single crystal blades for aircraft gas turbine engines.

Complex yielding behavior as a function of crystallographic orientation has been observed in single phase γ' and nickel-base superalloys containing a high volume fraction of the γ' phase^{1,2}. Slip on the cube rather than octahedral planes has been frequently observed for orientations near $[\bar{1}11]$. Also, even for orientations exhibiting normal octahedral slip, yield stresses have been found to deviate from Schmid's Law as a function of orientation and even differ in tension and compression.

In this study, the compressive yielding behavior of the single crystal superalloy René N4 has been studied and compared with previous data for yielding in tension³ and in fatigue⁴. Slip systems were identified by slip trace analysis. The orientation and tension-compression dependencies of yielding by octahedral slip has been numerically evaluated according to a model proposed by Lall, Chin, and Pope for a single phase γ' alloy⁵. We will refer to this as the LCP model for the sake of brevity.

The René N4 single crystals were provided by the General Electric Company, Aircraft Engine Group, Evandale, Ohio; Dr. P.K. Wright of that organization made many helpful comments.

MATERIALS AND TEST PROCEDURES

The composition of the master heat from which the crystals were cast

is presented in Table I together with the nominal composition and heat treatment of the alloy. The average size and volume fraction of the γ' in the René N4 studied are about $0.25\mu\text{m}$ and 65v/o.

The crystals were cast as slabs approximately $12 \times 5.5 \times 1.3 \text{ cm}$. Specimens were cut from material remaining from the previous studies. The crystallographic orientation of each specimen was determined by x-ray diffraction. An average orientation for each group of specimens is presented in Table II and graphically in Figure 1. Also presented in Table II are average values of the various Schmid factors to be considered. However, calculations were made using precise values determined for each specimen. In addition, the slabs from which the specimens were cut are indicated in Table II. Specimens with [001] compression axes were cut from all slabs where sufficient material remained to aid in assessing any variability from slab to slab.

The compression test specimens had 5mm square gage sections and were 10mm long. The surfaces of the specimens were polished with successively finer papers finishing with a $0.3\mu\text{m}$ diamond paper. The compression testing was conducted in an Instron testing machine using a fixture to convert separation of the crossheads into compression of the sample. A slurry of MoS_2 in mineral oil was used to lubricate the ends of the specimens. The tests were run in air at a constant crosshead speed. This resulted in initial strain rates which depended on crystal orientation. For all test temperatures, the initial strain rates ranged from about $12 \times 10^{-4} \text{ sec}^{-1}$ for the [001] orientation, which has the lowest elastic modulus, to about $5 \times 10^{-4} \text{ sec}^{-1}$ for the $[\bar{1}11]$ orientation, which has the highest. Temperature was controlled to $\pm 2^\circ\text{C}$. The test data were recorded as load versus crosshead displacement, and plastic strain obtained using

the offset from the machine-specimen elastic loading line and the specimen gage length. Tests were run until about 2% plastic strain was attained.

RESULTS AND DISCUSSION

Primary Cube Slip.- Results from the tensile tests performed previously³ will be discussed together with compression test results obtained herein without constant reference. On the figures each point indicates the orientation of the specimen and the type of test.

Where possible the operating slip system for each test was confirmed by slip trace analysis (and by tensile axis rotation for the tensile tests³). Since no slip traces were observed on specimens tested at 980°C and some tested at 870°C, identification of the slip system rests on finding consistency among calculated values of the CRSS on a particular slip system.

For specimen axes defined to be in the standard stereographic triangle, the primary cube slip system is (001)[$\bar{1}10$]. The orientations of specimens exhibiting cube slip and the values of the CRSS for cube slip obtained are shown as a function of temperature in Fig. 2. The [$\bar{1}11$] specimens, which had the highest Schmid factor for primary cube slip of all the specimens, exhibited cube slip at all temperatures, even room temperature. It may be seen that the range of orientations exhibiting cube slip expanded with increasing test temperature. Slip traces were observed on the [023] specimen tested at 870°C indicating the great extent of the orientations exhibiting cube slip at that temperature. It may be seen in Fig. 2 that consistent values of the CRSS on the cube slip system were obtained at 870 and 980°C for the [$\bar{1}11$], [$\bar{1}12$], [$\bar{2}36$], [$\bar{1}67$], and [023] specimens.

Both the results shown here and the values of yield strength obtained from low cycle fatigue tests⁴ show a tension-compression anisotropy in yielding by primary cube slip at high temperatures. The CRSS for cube slip is higher in compression than in tension at 760°C, and the opposite at 980°C. The same behavior can be observed in the alloy PWA 1480 studied by Shah and Duhl². Discussion of this subject will be deferred until after introduction of the LCP model.

Octahedral Slip.- The orientations exhibiting octahedral slip and the values of the CRSS for octahedral slip obtained are shown as a function of temperature in Fig. 3. Again, the slip system has been confirmed either by slip trace analysis, tensile axis rotation, or for some of the 870 and 980°C tests, by comparison of calculated values of the CRSS on the octahedral slip system.

First it should be pointed out that there is little difference among the slabs from which the specimens were cut. [001]-oriented specimens cut from 5 of the 8 slabs were tested in compression at both 760 and 980°C. The slabs from which specimens were cut are identified in Table II. It may be seen in Fig. 3 that agreement among the [001] compression tests is very good at 760°C. The specimen from slab I appears somewhat weaker than the others, however no specimens with other orientations were cut from this slab. Agreement at 980°C is also good generally, however the [001] and $[\bar{1}26]$ specimens from slab A, and the [001] specimen from slab I appear somewhat stronger than the other compression specimens. In addition to the appearance that differences among the slabs were small, any systematic effect upon the results due to differences was reduced by cutting specimens from two of the slabs, A and C, that had orientations at opposite sides of the stereographic triangle. See Table II.

We will next consider tests performed at temperatures up to 760°C. A tension-compression anisotropy which depends on crystal orientation may be seen in the 760°C data, Fig. 3. This tension-compression anisotropy is similar to that observed for octahedral slip in some single phase γ' alloys⁷ and another single crystal superalloy². The [001] orientation is stronger in tension than in compression, and the [011] orientation shows the opposite behavior.

Since the same tension-compression anisotropy of yielding in Rene N4 was also observed in push-pull fatigue tests⁴ it is felt that there was little effect, if any, of the different test methods employed in the tension and compression tests. That is, there was little effect of any lateral end constraint in the compression tests, as has been observed in some tests of single crystals⁸.

The increasing strength of γ' with increasing temperature is generally thought to be produced by a thermally activated process wherein the lead dislocation of the superdislocation pairs on the octahedral slip plane cross slips onto the cube cross slip plane becoming immobilized. The LCP model (which is an extension of the model of Takiuchi and Kuramoto⁹) considers the individual steps in this process and how they might be affected by the applied stress. The model considers that the dislocation is initially dissociated on the octahedral plane and must be constricted before it can cross slip. The applied stress may aid both this constriction and the cross slip event. However, the dependence of these processes on the orientation of the applied stress is not the same as that for dislocation motion on the octahedral plane.

The model adds additional terms to Schmid's Law as shown:

$$S_1 \sigma_y = T_n + A_0 \exp[(-H_0 + A_2 S_2 \sigma_y + \delta A_3 S_3 \sigma_y)/RT], \quad (1)$$

where the Schmid factors $S_1 = S_{(\bar{1}11)[\bar{1}01]}$,

$$S_2 = S_{(010)[\bar{1}01]}$$

$$S_3 = S_{(\bar{1}11)[1\bar{2}1]}$$

T_n is analogous to the normal CRSS for octahedral slip at a given temperature, σ_y is the yield stress, H_0 is the activation energy representing the natural tendency for cross slip onto the cube plane to lower the antiphase boundary energy, A_1, A_2 , and A_3 are constants, and δ is +1 for tensile loading and -1 for compressive loading. The equation represents Schmid's Law with the addition of an extra term. The additional term has been written as a combined thermally activated process, since the deviations from Schmid's Law are observed to increase with increasing temperature up to the peak strength temperature. The exponential term subscripted with 2 represents the applied stress resolved onto the cube cross slip plane, and that subscripted with 3 represents the component acting on the initial dissociated lead dislocation on the octahedral plane. The δ expresses the opposite effect of tensile and compressive loading on constriction of the dissociated dislocation on the octahedral plane.

In order that the present data can be precisely evaluated in terms of the above model it has been approximated in a linear form so that linear

regression analysis may be applied. If, in Eqn. 1, the exponent of $\exp[(A_2 S_2 \sigma_y + \delta A_3 S_3 \sigma_y)/RT]$ is small, it may be approximated as $[1 + (A_2 S_2 \sigma_y + \delta A_3 S_3 \sigma_y)/RT]$. After rearrangement of terms the variables are separated in the form

$$1/\sigma_y = b_1 S_1 + b_2 S_2 + \delta b_3 S_3, \quad (2)$$

where

$$b_1 = 1/[T_n + A_0 \exp(-H_0/RT)],$$

$$b_2 = (-b_1 A_0 A_2 / RT) \exp(-H_0/RT),$$

and

$$b_3 = (-b_1 A_0 A_3 / RT) \exp(-H_0/RT).$$

In this form, the orientation dependence of σ_y at a given temperature can be numerically evaluated by linear regression analysis.

Figs. 4 and 5 show the observed yield strengths vs. those predicted by both Schmid's Law and the LCP model for room temperature and 650°C. These same two predictions for 760°C are presented separately in Figs. 6(a) and (b). The symbol code is consistent in indicating for each point what orientation is represented, whether the test was in tension or compression, and which model is represented. The points with a 'c' beside them indicate an orientation exhibiting cube slip. These orientations were not considered in establishing the regression equation based on the LCP model, but their yield strengths on the octahedral slip system have been predicted using that model.

It is readily apparent that the LCP model provides an improved fit over Schmid's Law, which may be no surprise since the model contains two more parameters. Further examination is necessary before commenting on

the apparent validity of the model as applied to a nickel-base superalloy. The values of the coefficients in the LCP model as determined by regression analysis together with the T-ratios for each are presented in Table III. A T-ratio of about 1.7 or greater is generally taken to indicate that a term is statistically significant, that is, nonzero.

It may be seen that the b_2 term is the least important of the two terms representing the deviations from Schmid's Law. Again, this term addresses the effect of the applied stress on driving the lead dislocation off onto the cube cross slip plane. This source of deviation from Schmid's Law was the first proposed and does explain the type of deviations from Schmid's Law observed in some $L1_2$ compounds⁷. However, not only is this term not very important in describing the behavior of the nickel-base superalloy studied here, it is of the wrong sign. For room temperature and 650°C this term cannot be said to be non zero. However, the 760°C data appear to show that there is a slight decrease in yield strength with increasing stress on the cube cross slip system.

The b_3 term accounts for most of the observed Schmid's Law deviations. It is larger, significant, and has the expected sign, as compared to the b_2 term. This, again, is the term dealing with the tension-compression anisotropy. Even for the tests at 650°C, where only compression tests were conducted, a T-ratio of 1.6 is obtained for the value of b_3 .

Shah and Duhl² cited the LCP model as qualitatively explaining the orientation dependence of yielding by octahedral slip in the nickel-base superalloy which they studied, PWA 1480, but did not comment on the relative importance of the two terms leading to deviations from Schmid's Law. A comparison with René N4 may be made at 650°C. Analysis of their

data for the [001] and [011] orientations also shows that the b_2 term has little significance. Their yield strength values are predicted within 4% by a model which only adds the third term of the LCP model to Schmid's Law. The regression fit equation is $1/\sigma_y = .00222S_1 - \delta(.000298)S_3$.

Thus, for René N4 and PWA 1480, the effect of the stress on the cube cross slip system is small. The stress tending to constrict or expand the dissociated dislocations on the octahedral plane is most important in explaining the observed Schmid's law violations. In fact, the value of b_3 obtained for PWA 1480 is very nearly the same as that obtained for René N4.

It should be pointed out that deviations from Schmid's Law are not the rule in nickel-base superalloys. From a review of the literature prior to 1984 one would conclude that nickel-base superalloys exhibit little deviation from Schmid's Law¹. A systematic study of the effects of alloy composition on deviations in yielding behavior from Schmid's Law is needed, and might yield very useful basic information about the mechanisms of strengthening of the γ' phase through interpretation by a model such as that of Lall, Chin, and Pope.

Above 760°C, which is about the peak strength temperature in Rene N4, deviations from Schmid's Law are reduced and the orientation and tension-compression dependencies of yield strength for octahedral slip change (Fig. 2). At least for 980°C, for which both tensile and compressive yield strengths were obtained, it appears that yield strengths are somewhat higher in compression than in tension for the orientations exhibiting octahedral slip. This may be seen both in Fig. 2, and the yield strengths obtained previously from fatigue tests⁵. Alloy PWA 1480 appears to show almost no deviation from Schmid's Law or tension-

compression anisotropy at high temperatures².

It appears in Fig. 3 that the [001] orientation may be stronger than the [011], at least in compression, at 870 and 980°C. However, this was not observed in the earlier fatigue testing. Thus, we cannot conclude whether there is any deviation from Schmid's Law at the higher test temperatures. Certainly if there is, it is much smaller than at 760°C.

Now that the LCP model has been introduced, we would like to return to the question of tension-compression anisotropy for cube slip. It has been proposed⁶ that dislocations on the cube plane in γ' dissociate into partials on the octahedral cross slip plane. If so, the resolved stress acting to constrict the partials should aid slip on the cube plane, the opposite of the case for octahedral slip. Thus, we might expect to see the behavior observed at 760°C. The orientations exhibiting cube slip appear to be stronger in tension than in compression, the opposite behavior of the other orientations near the $[\bar{1}11]$ -[011] border which exhibit octahedral slip. However, no suggestion is offered for why this tension-compression anisotropy for cube slip reverses itself at 980°C.

Another similarity between between cube and octahedral slip behavior is the peak strength near 760°C. This also suggests that a thermally activated locking mechanism is appropriate for dislocations on the cube plane as well as for those on the octahedral plane.

SUMMARY

Compression tests at temperatures from room temperature to 980°C were performed on single crystals of the nickel-base superalloy René N4 with various orientations. Compressive yield strengths were compared with

tensile yield strengths obtained earlier and analysed with respect to their dependence on temperature, orientation, and tensile or compressive stress. The following results and conclusions were obtained:

1. Primary cube slip occurred for orientations near $[\bar{1}11]$ at all temperatures. The range of orientations exhibiting cube slip increased with increasing temperature, until at 980°C only orientations very near the $[001]$ or $[011]$ exhibited octahedral slip.
2. The critical resolved shear stress for cube slip exhibited a peak near 760°C , similar to that observed for octahedral slip.
3. Tension-compression anisotropies existed for both octahedral and cube slip.
4. Deviations from Schmid's Law for orientations exhibiting octahedral slip could be explained by the model of Lall, Chin, and Pope. An orientation dependent tension-compression anisotropy was the major deviation from Schmid's Law. This is explained in the model as the effect of the applied stress in promoting constriction of the dissociated dislocations on the octahedral plane prior allowing immobilization by cross slip onto the cube plane.
5. The effect of the applied stress in promoting cube cross slip, which would increase the strength of orientations away from the $[001]$, was found to be relatively unimportant in René N4. Analysis of published data for alloy PWA 1480 showed the same result.

REFERENCES

1. D.P. Pope, and S.S. Ezz: Int. Met. Rev., 1984, vol. 29, pp. 136-167.
2. D.M. Shah, and D.N. Duhl: in Superalloys 1984, M. Gell, et al., eds., pp. 105-114, Metallurgical Society of AIME, Warrendale, PA, 1984.
3. R.V. Miner, R.C. Voigt, J. Gayda, and T.P. Gabb: (submitted to Metallurgical Transactions A.)
4. T.P. Gabb, J. Gayda, and R.V. Miner: (submitted to Metallurgical Transactions A.)
5. C. Lall, S. Chin, and D.P. Pope: Metall. Trans. A., 1979, vol. 10A, pp. 1323-32.
6. B.H. Kear, and H.G.F. Wilsdorf: Trans. Am. Inst. Min. Metall. Pet. Eng., 1962, vol. 224, pp. 382-86.
7. S.S. Ezz, D.P. Pope, and V. Paidar: Acta Metall., 1982, vol. 30, pp. 921-26.
8. C.C. Law, A.F. Giamei, and A.V. Karg: Mater. Sci. Eng., 1977, vol. 29, pp. 29-35.
9. S. Takeuchi, and E. Kuramoto: Acta Metall., 1973, vol. 21, pp. 415-25.

TABLE I. Composition and Heat Treatment of the René N4 Crystals Studied. Balance of composition is nickel.

| | Element, w/o | | | | | | | | | | |
|---------|--------------|------|------|-----|------|------|------|------|-------|------|------|
| | Al | Ti | Ta | Nb | Cr | Mo | W | Co | C | B | Zr |
| Nominal | 3.7 | 4.2 | 4.0 | 0.5 | 9.0 | 1.5 | 6.0 | 7.5 | - | - | - |
| Actual | 3.77 | 4.24 | 3.96 | 0.5 | 9.26 | 1.60 | 5.88 | 7.53 | 0.005 | 0.01 | 0.01 |

Heat Treatment: 1260°C/2h/gas quench + 1080°C/4h/air cool + 900°C/16h/air cool.

TABLE II. Orientations of the René N4 Crystals Studied.

| Symbol | Approx. Orient. | Castings | Average Measured Orientations | | | Average Schmid Factors | | | |
|--------|-----------------|-----------|-------------------------------|-----|---|------------------------------------|--------------------------|--|--------------------------|
| | | | | | | S ₁ ' | S ₂ ' | S ₃ ' | S ₄ ' |
| | | | h | k | l | ($\bar{1}11$) [$\bar{1}01$] | (010) [$\bar{1}01$] | ($\bar{1}11$) [$\bar{1}\bar{2}1$] | (001) [$\bar{1}10$] |
| □ | [001] | A,E,G,I,J | -0.02 | .04 | 1 | .425 | .030 | .021 | .040 |
| D | [$\bar{1}26$] | A | -.15 | .31 | 1 | .490 | .230 | .055 | .290 |
| ◇ | [$\bar{1}12$] | C | -.53 | .60 | 1 | .410 | .400 | -.120 | .490 |
| ◇ | [$\bar{2}36$] | J | -.38 | .48 | 1 | .455 | .350 | -.070 | .425 |
| △ | [$\bar{1}11$] | A | -.87 | .96 | 1 | .300 | .465 | -.155 | .480 |
| ◻ | [$\bar{1}67$] | L | -.13 | .35 | 1 | .460 | .390 | -.190 | .400 |
| ◊ | [011] | C | -.18 | .96 | 1 | .440 | .440 | -.230 | .415 |
| ◊ | [011] | D,E | -.09 | .98 | 1 | .420 | .385 | -.255 | .380 |
| ○ | [023] | G | -.03 | .67 | 1 | .475 | .340 | -.105 | .340 |

TABLE III. Coefficients in the model, Eqn. 2; their T -ratios; n , the number of observations; and S , the standard deviation of the dependent variable.

| TEMPERATURE, °C | $b_1 \times 10^3$, MPa | T - Ratio | $b_2 \times 10^3$, MPa | T - Ratio | $b_3 \times 10^3$, Pa | T - Ratio | n | $S \times 10^5$, MPa |
|--------------------|----------------------------|----------------|----------------------------|----------------|---------------------------|----------------|-----|--------------------------|
| RT | 2.47 | 27 | .014 | .1 | -.300 | 2.9 | 13 | 6.1 |
| 650 | 2.74 | 23 | .180 | .8 | -.280 | 1.6 | 6 | 1.7 |
| 760 | 2.56 | 40 | .345 | 3.6 | -.727 | 9.6 | 18 | 5.6 |

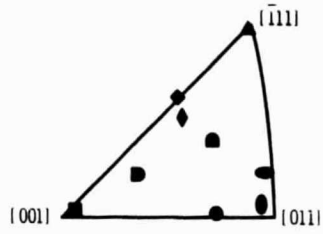


Figure 1. - Orientations of the crystals studied.

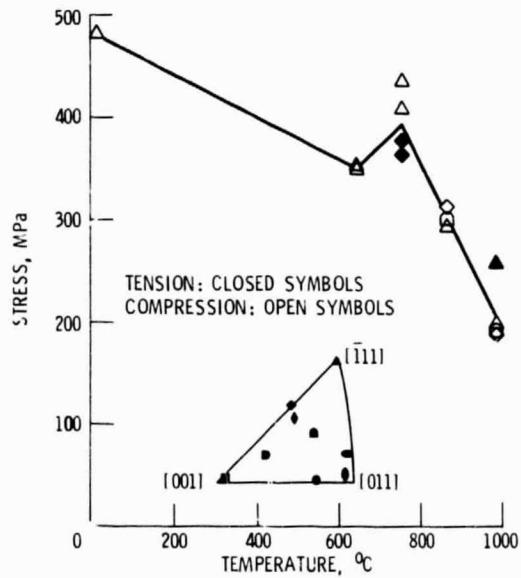


Figure 2. - Temperature dependence of the CRSS for cube slip in Rene N4 assuming Schmid's Law.

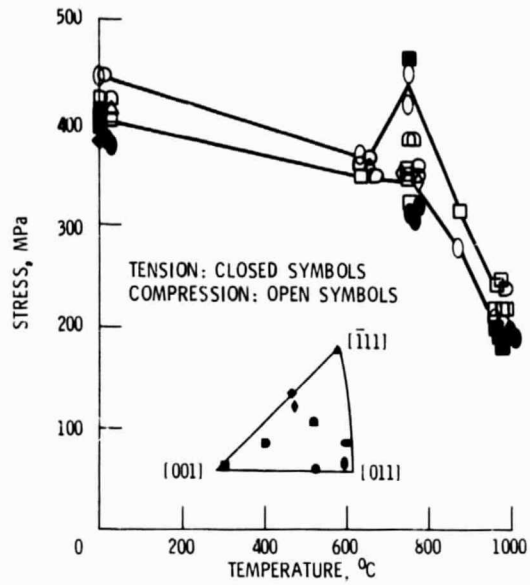


Figure 3. - Temperature dependence of the CRSS for octahedral slip in Rene N4 assuming Schmid's Law.

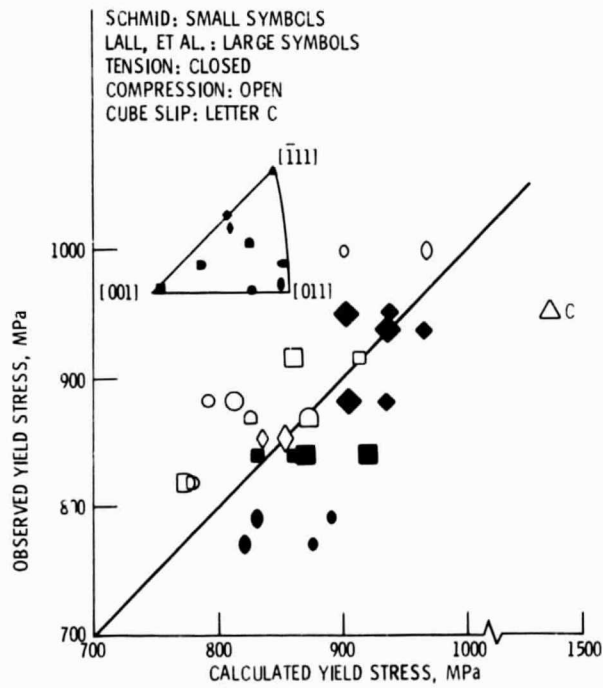


Figure 4. - Observed vs. calculated values of tensile and compressive yield strengths for octahedral slip as a function of orientation for Rene N4 at room temperature using Schmid's Law or the model of Lall, Chin, and Pope.

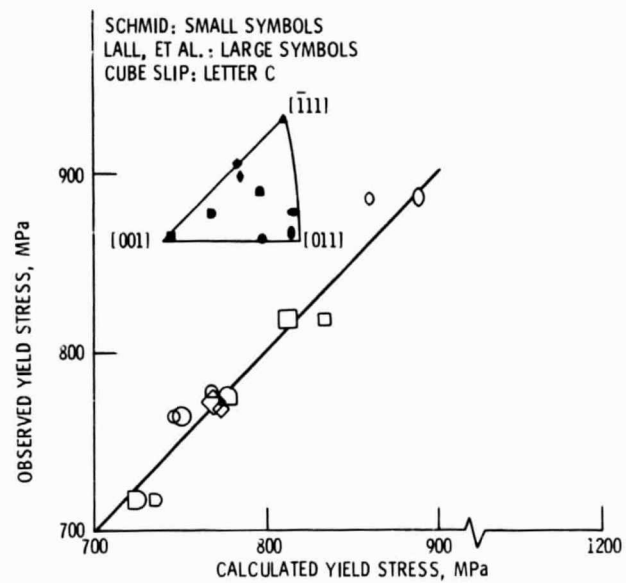


Figure 5. - Observed vs. calculated values of compressive yield strength for octahedral slip as a function of orientation for Rene N4 at 650°C using Schmid's Law or the model of Lall, Chin, and Pope.

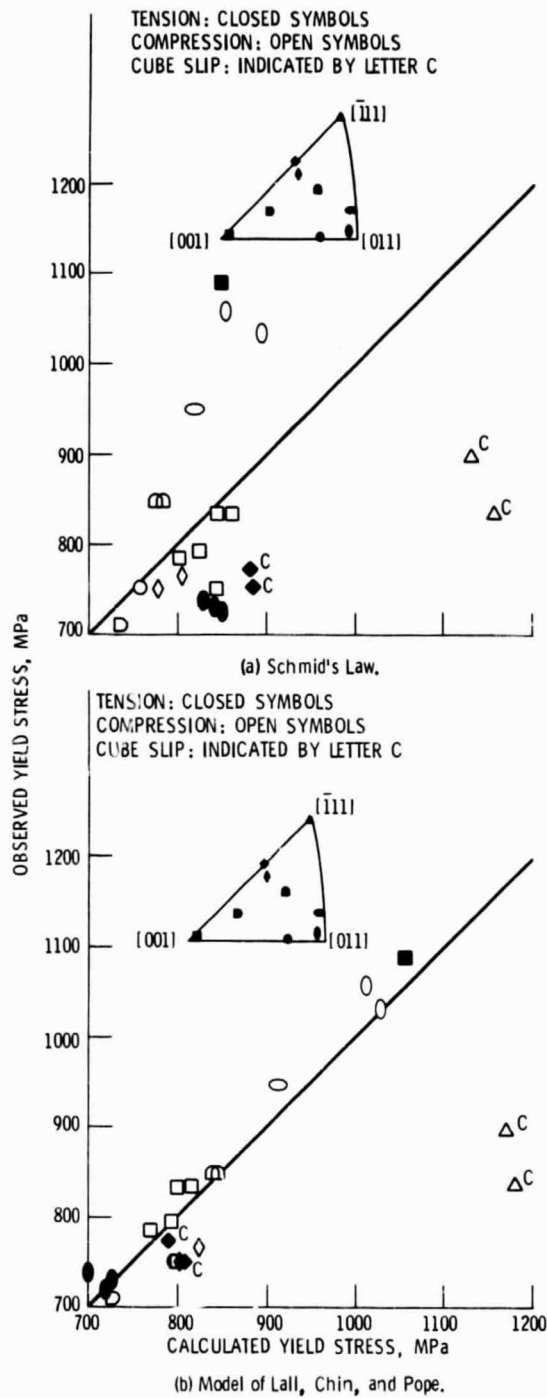


Figure 6. - Observed vs. calculated values of tensile and compressive yield strengths for octahedral slip as a function of orientation for Rene N4 at 760 °C.