DISPERSION STRENGTHENED NICKEL-YTTRIA SHEET ALLOY PRODUCED FROM COMMINUTED POWDERS

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ABSTRACT

An investigation was conducted to determine whether a nickel matrix with yttria as a dispersoid could be produced by a comminution and blending (wet attrition-NASCAB) approach. Concentration of yttria, powder cleaning temperature, screening (sieving) of the powders, and amount of thermomechanical working were major variables. Tensile strength and stress-rupture life at 1093°C were determined. A product containing 4v/o Y₂O₃, cleaned at 315 or 371°C with screening exhibited 1093°C tensile strength equivalent to NASCAB Ni-4ThO₂ and to commercially produced thoriaed nickel sheet.
This study reports on initial efforts to dispersion strengthen nickel with Y2O3 in an attempt to replace radioactive ThO2 as the strengthening phase in dispersion strengthened alloys. Nickel-Y2O3 powders were processed by the NASA comminution and blending (NASCAB) method and subsequently thermomechanically worked. Experimental variables included volume percent Y2O3 (2% and 4%), powder cleaning temperature (315, 371, and 426°C), a screening step in the process, and the number (up to 23) of cold-roll-anneal cycles. Tensile strengths, determined at 1093°C, as well as some stress-rupture life data are presented.

This study demonstrated that nickel with 4% Y2O3 with good 1093°C mechanical properties could be produced by the NASCAB process. The 4% Y2O3 composition was stronger than that containing 2% Y2O3. The 315 and 371°C cleaning temperatures were equally effective in promoting strength while the 426°C cleaning temperature resulted in lower strength. Powders which were screened during processing generally yielded higher strengths than those which were not screened. Although the screening was beneficial, it tended to increase the scatter in the data. There is some indication that maximum strength can be obtained at both an intermediate number (10) of cold-roll-anneal cycles and also at a larger number (21) of cycles. A strength level of 124.1 MN/m² (18,000 psi.) at 1093°C was readily obtained. This compares favorably to Ni-4% ThO2 made by the same process and to commercial TD-Ni sheet. The stress-rupture results at 1093°C are comparable to the data reported for commercial TD-Ni sheet at 1093°C.
INTRODUCTION

The high temperature long time strength properties of some materials can be improved by the addition of a fine stable oxide dispersoid, ref. 1. Work in this field has been going on over 20 years and currently some dispersion strengthened materials are being considered for gas turbine engine applications at temperatures to about 1100°C. Early work in the field was done using Al₂O₃ (alumina) as the dispersoid; however, in later studies ThO₂ (thoria) was used more extensively. Although ThO₂ has excellent high temperature stability in such alloys it does have the disadvantage of being radioactive. The ThO₂ is primarily an alpha emitter with a relatively slight amount of gamma radiation.

There are a number of reasons why companies are reluctant to use material strengthened with ThO₂ even though a small amount is used. For one thing, material of this nature requires maintaining a safety engineer to monitor its use and imposes restrictive requirements in handling and fabrication. Also, scrap thoriated material should be isolated for disposal. Failure to do this could lead to the introduction of radioactive material into metal scrap material. Thus, interest at NASA-Lewis has centered on replacing ThO₂ with a nonradioactive oxide dispersoid such as Al₂O₃ and Y₂O₃ (yttria) in the comminution and blending process for producing dispersion strengthened alloys. Initial efforts with Al₂O₃ were reported in ref. 2. This paper presents the results of a preliminary study to use Y₂O₃ as a strengthening dispersoid in nickel.

The first successful commercial nickel base dispersion strengthened products were made using a complex chemical coprecipitation approach with ThO₂ (ref. 1). Recently other dispersion strengthening studies, employing a dry attrition (mechanical alloying) approach, have used Y₂O₃ as the dispersoid. Currently, commercial alloys using Y₂O₃ as the strengthening dispersoid are available, ref. 3, or nearly ready for production, ref. 4. Efforts as NASA-Lewis have concentrated on producing fine oxide dispersions in metal by a wet attrition mechanical comminution and blending approach known as NASCAB.

In the NASCAB process a metal such as nickel and a stable oxide are comminuted and blended in an attritor with an organic fluid to produce exceedingly fine powders on the order of 200⁰ A. The fine powders are precleaned to reduce any matrix oxide and to anneal the powders prior to cold compaction. The compacted slabs are sintered and then subjected to hot rolling followed by cold rolling, with intermediate anneals. Using this method dispersion strengthened Ni-ThO₂ sheet material with excellent high temperature properties have been produced, ref. 5. In a recent study, ref. 2, some dispersion strengthened Ni-Al₂O₃ sheet material was produced which had 1093⁰ C tensile properties comparable to TD-Ni (commercial chemically produced Ni-ThO₂) sheet material.
In these previous studies (refs. 2 and 5) some scatter in the data was thought to be associated with two screening steps used in the process. It appeared possible that the screening prior to and after precleaning might be resulting in some segregation of the fine dispersed particles thus negating the beneficial effects of the attriting process.

The object of this study was to determine the feasibility of producing a Ni-Y$_2$O$_3$ composition with mechanical properties comparable to TD-Ni* and NASCAB Ni-ThO$_2$ by applying the wet attriting (NASCAB) process. The major variables were concentration of Y$_2$O$_3$ (2 and 4 volume percent), powder precleaning temperature (315°, 371°, and 426° C), the number of cold-roll-anneal cycles in the thermomechanical processing, and the use or absence of the powder screening (sieving) steps. The effectiveness of these variables in producing a product comparable to TD-Ni and NASCAB Ni-ThO$_2$ were judged by the 1093° C tensile properties and stress-rupture behavior.

**MATERIALS**

The raw materials used in this investigation consisted of 99.7% pure Inco Type 287 carbonyl nickel powder with an average particle size of 2.5 micrometers and yttrium oxide powder with an average particle size of 3.3 micrometers.

**APPARATUS AND PROCEDURE**

The process used to produce the Ni-Y$_2$O$_3$ sheet material in this study is outlined in the flow chart in figure 1. A triple stirrer attritor was used in this investigation as described in ref. 2. After drying the slurry from the attritor a breakable cake remains. This cake was then either precleaned or passed through a 40 (420 µm) mesh screen prior to precleaning. Precleaning, as used in this report, means heating the powders in H$_2$ prior to compaction and serves a two-fold purpose. The first is to reduce any matrix oxide in the starting material or that may have formed during the grinding. The second purpose is to anneal the powder to aid in compaction into slabs. Previous experience (refs. 2 and 5) has shown that heating at 315° C was effective in achieving good properties; however, it was felt that heating at higher temperatures might help to improve the properties of the materials by virtue of a more effective reduction of matrix oxides. The precleaning procedure described in ref. 2 was modified here to the extent that two additional temperatures were evaluated as to effectiveness in improving properties. From fig. 1 it may be seen that a grind of nickel plus Y$_2$O$_3$ was divided so that 1/2 was screened

*TD-Ni was chosen as a commercial, well documented, standard for comparison when this study was initiated; however, it is no longer available commercially.*
and the remainder was not. In each case then the cake was again di-
vided so that approximately 1/3 was precleaned at each of the indicated 
temperatures in a hydrogen atmosphere followed by a vacuum treatment 
as described in ref. 2. Material that had been screened prior to pre-
cleaning was again screened after precleaning whereas the remaining 
material received no screening treatments. A two step compaction 
procedure was used for all material which consisted of die pressing to 
shape the slab and isostatic hydraulic pressing to provide some den-
sification and green strength (ref. 2). The green slabs were then 
heated in a hydrogen atmosphere at a nominal heating rate of 40° C/hour 
until 1093° C was reached. The heating was interrupted when the 
moisture in the effluent gas exceeded 100 ppm and temperature was held 
constant until the moisture level fell below 100 ppm. Heating was re-
sumed when effluent gases contained less than 100 ppm moisture and 
continued until 1093° C was reached. All slabs were then held at 
1093° C for two hours. After such a heating and sintering treatment 
slabs were in the range of 80-85% of theoretical density based on the 
weight and physical dimensions of the slab.

Hot compaction to densify the slabs was accomplished by hot 
rolling from a hydrogen atmosphere furnace at 1093° C (ref. 2). Each 
slab was reduced a total of 50% in thickness by two hot passes (30% 
reduction per pass). At this point the nominal dimensions of a slab 
were approximately 0.2 cm x 1.5 cm x 10 cm (0.080 in. x 0.60 in. 
× 4.0 in.).

Further working was done at room temperature for the desired 
number of cold roll anneal cycles. A cold roll anneal cycle consisted 
of reducing the slabs 10% prior to annealing in a hydrogen atmosphere 
at 1200° C for 1/2 hour.

Specimens for tensile testing and metallographic evaluation were 
sheared from the sheet at the desired number of (cold-roll-anneal) 
cycles.

Specimens were tensile tested in a conventional screw driven 
machine at a crosshead speed of 0.05 cm/min. at 1093° C in a vacuum of 
10^{-5} torr. Stress-rupture life was determined at 1093° C in a vacuum 
of 10^{-5} torr using a lever arm beam.

Conventional metallographic techniques were used to evaluate the 
materials being studied. These included light optical microscopy and 
both replication and transmission electron microscopy.

RESULTS AND DISCUSSION

Effect of Composition

Two compositions of Ni-Y2O3 were produced in this investigation: 
Ni + 2 volume percent Y2O3 and Ni + 4 volume percent Y2O3. Both alloys
were precleaned at 315° C and were produced with and without the screening steps in the process. Results of the 1093° C tensile tests are shown in Table I and figure 2. Ultimate tensile strength is plotted against the number of cold-roll-anneal cycles. The curves are based on the calculated values resulting from a least mean square computation. Included in figure 2 is the reported range of typical strength values at 1093° C for commercial TD-Ni (ref. 6) and data for NASCAB Ni-4ThO₂ sheet material as reported in ref. 5 using screening in the process.

It can be seen in figure 2 that the 4v/o Y₂O₃ alloy is much stronger than the 2v/o Y₂O₃ alloy in both the screened and unscreened conditions. In comparing the relative 1093° C tensile strength of the two Y₂O₃ alloys with TD-Ni it is apparent that the 2v/o Y₂O₃ alloy is lower whereas the 4v/o Y₂O₃ alloy is in the same range of strength as TD-Ni. However, in the case of the NASCAB Ni-ThO₂ it is seen that the curve is generally above the Ni-4Y₂O₃ curve. The scatter at 21 cycles is large, however it appears that the Ni-4Y₂O₃ material has the potential of attaining high strength. Maximum strength was attained for both materials when 21 cold-roll-anneal cycles were applied during processing. While it was established that the Ni-4Y₂O₃ has better 1093° C tensile strength than the Ni-2Y₂O₃, further study would be required to determine the optimum dispersoid concentration.

**Effect of Processing Variables**

**Precleaning temperature.** Only the 4v/o Y₂O₃ material was subjected to several precleaning temperatures. The results of the 1093° C tensile tests for the 4v/o Y₂O₃ material precleaned at 315° C, 371° C, and 426° C are presented in Table I and figure 3. Ultimate tensile strength is plotted against the number of cold-roll-anneal cycles and the curves are based on the calculated values resulting from a least mean square computation. Data are presented for both the screened and unscreened condition at each precleaning temperature.

On the basis of the tensile strength it appears that, the 315° C and 371° C precleaning temperatures were effective in producing good properties. The 426° C precleaning temperature resulted in lower strength. It is quite possible that at 426° C the fine Y₂O₃ particles in the undensified powder begin to agglomerate and therefore the strength of the material was reduced.

**Screening versus not screening.** Some concern was present in previous studies (refs. 2 and 5) that the screening of blended matrix and oxide powders would cause "unblending" or segregation of the matrix and oxide. If this did occur it could result in a decrease in mechanical properties of the dispersion material. Although the results obtained with and without screening are not as well defined as those obtained for differences in dispersoid concentration, the evidence in this study indicates that, in general, screening is not detrimental to
the mechanical properties. In fact, the screened material is generally stronger than the unscreened material, (fig. 3) however, an increased scatter in the data was noted for the screened material.

Thermomechanical processing (TMP). The effect of the cold working of the Ni-4Y2O3 material is shown in figure 3. For the unscreened material the response to TMP appears to be similar for the 315° C preclean temperature and the 426° C preclean temperature. That is in both cases the curves are relatively flat, although the 315° C curve is at a slightly higher level than the 426° C curve. However, the tensile strength increases with TMP for the unscreened material precleaned at 371° C to about 17 cold-roll-anneal cycles then levels off.

The screened material responds to TMP quite differently from the unscreened material at 315° C and 371° C as shown in figure 3. For example, in contrast to the relatively flat response noted for the unscreened material, the screened material precleaned at 315° C increased in tensile strength with increased number of cold-roll-anneal cycles. In the case of the material precleaned at 371° C the tensile strength of the screened material was much higher than the unscreened material at 7 cold-roll-anneal cycles. However, with increased working the screened material decreased in strength so that at 21 cycles the properties are practically the same. When the powders were precleaned at 426° C both the screened and unscreened responded similarly in that both curves are relatively flat with the screened material at a slightly higher level. It should be noted that the 426° C precleaning temperature yielded the poorest properties.

It was shown earlier that the NASCAB Ni-4ThO2 material generally resulted in superior strength to the Ni-4Y2O3 material precleaned at 315° C. However, for the material precleaned at 371° C it may be seen that the yttriated material is comparable in strength to the thoriated material at a low number of cold-roll-anneal cycles, but with further working the yttriated material decreases in strength whereas the thoriated material increases. Although the data at 10 cold-roll-anneal cycles show a wide scatter they do indicate a potential for good strength which is comparable to the NASCAB Ni-4ThO2.

In view of the fact that relatively good strength is obtained at 371° C with minimal cold working (10 cold-roll-anneal cycles), this treatment of screened material would yield a good practical compromise in terms of strength obtainable versus the amount of processing.

Effect of Processing Variables on Tensile Elongation, Stress-Rupture, and Microstructure

Tensile elongation at 1093° C. The results of elongation measurements from the 1093° C tensile tests are shown in Table I.
Elongation was measured from the recorder chart based on the crosshead speed and chart speed. Post-test elongation measurements of failed specimens could not be made due to the irregular fracture of many of the specimens. There does not appear to be any strong correlation between any processing variable and elongation.

The reported 1093°C tensile elongation for TD-Ni ranged from 5 to 9 percent. From Table I it may be seen that the 1093°C tensile elongation for the Ni-4v/o Y2O3 material ranged from 3.9 to 27.5 percent with about 2/3 of all the values in excess of 9 percent. Elongation data for the NASCAB NiThO2 material are not available.

Stress-rupture results at 1093°C. Limited stress-rupture testing was done with the few specimens available which were chosen as close as possible to a constant number of cold-roll-anneal cycles. Any effect of this variable (number of cold-roll-anneal cycles) on the stress-rupture behavior is not apparent from these tests. Since the 4v/o Y2O3 material was superior to the 2v/o Y2O3 material in 1093°C tensile strength, only the 4v/o Y2O3 alloy was tested in stress rupture.

These specimens were tested in stress-rupture at 1093°C with a stress of 51.7 MN/m² (7.5 ksi) or 68.9 MN/m² (10.0 ksi). The stress rupture results are presented in Table II and figure 4. Since the supply of specimens was limited, it is difficult to see trends in stress-rupture life as a function of processing variables.

Although wide scatter in the stress-rupture data was observed, it should be noted that the results encompass the reported range of rupture life for commercial TD-Ni. There are not sufficient stress-rupture data for NASCAB NiThO2 to allow a comparison to be made.

Microstructure. Electron micrographs of specimens were examined after rolling in order to compare the 2v/o Y2O3 material to the 4v/o Y2O3 material. Also, replicated microstructures of samples of the 4v/o Y2O3 material which showed a wide variation in tensile strength were examined. Typical microstructures of both the 2v/o Y2O3 and 4v/o Y2O3 materials are shown in figure 5(a, b, and c).

It may be seen from the micrographs that the specimen with the highest tensile strength, figure 5(a), has a dense uniform distribution of fine oxide particles. Also, elongated sub-grains may be seen throughout the structure. The micrograph of the 4v/o Y2O3 specimen with the lower tensile strength, figure 5(b), shows a fairly large number of very fine round oxide particles in the background; however, the elongated sub-grains seen in figure 5(a) are not apparent here. Also, a fairly large number of large elongated particles are present and some of these apparently have broken away from the matrix. Areas of deformed matrix at the edge of the elongated particles are also seen. This latter effect due to rolling has been noted previously,
ref. 7. Frequently, the cracks and deformed areas heal during annealing. Nevertheless, the presence of many large particles could interfere with the development of the microstructure which contributes to the strength of the material and could account for the low tensile strength observed for this material. It is interesting to note that no cracks or areas of deformed matrix are evident in the high strength material seen in figure 5(a).

The features seen in the micrograph in figure 5(b) are further accentuated in the 2v/o Y2O3 material shown in figure 5(a) which also had low strength. Here a much smaller number of fine particles is evident and more elongated and large particles which have broken away from the matrix may be seen. Thus there does appear to be some correlation of high temperature tensile strength and observed microstructural features such as size and distribution of the oxide particles and the presence of elongated sub-grains.

Examination of these materials using the light microscope and conventional metallographic techniques did not reveal the grain size or any significant aspects of the microstructure.

SUMMARY OF RESULTS

This investigation was conducted to determine the feasibility of using Y2O3 to replace ThO2 as the dispersoid in a nickel matrix (by wet attriting) and achieve properties comparable to TD-Ni and NASCAB Ni-4ThO2. Two concentrations of Y2O3 were studied. In addition, the effect of processing variables such as the use of screening and modified precleaning schedules applied to the powders prior to compaction on the properties of these materials was determined. The following results were obtained:

1. When using the NASCAB process, Y2O3 as a dispersoid in a nickel matrix is a suitable oxide addition to promote dispersion strengthening. A potential for good high temperature (1093° C) tensile strength was demonstrated.

2. Maximum tensile strength achieved with Ni-4Y2O3 was comparable to NASCAB Ni-4ThO2 and TD-Ni.

3. Material produced with 4 volume percent Y2O3 was superior to that produced with 2 volume percent Y2O3. The average 1093° C tensile strength was about 127.6 MN/m² (18.0 ksi) for the nickel plus 4 volume percent Y2O3 and about 54.5 MN/m² (7.9 ksi) for the nickel plus 2 volume percent Y2O3.

4. Tensile strengths of materials produced from powders pre-cleaned at 315° and 371° C were comparable and were higher than those pre-cleaned at 426° C.
5. Screening of the powders prior to compaction appears to be beneficial for the 1093° C tensile strength. Unscreened materials gave 1093° C tensile strengths about 9 MN/m$^2$ (1.3 ksi) lower than screened material.

6. Stress rupture life at 1093° C for the Ni-4Y$_2$O$_3$ material was comparable to TD-Ni.

CONCLUDING REMARKS

This study has shown that nickel can be effectively dispersion-strengthened by yttria when the material is processed by a communication and blending technique (NASCAB).

On the basis of this investigation it was determined that good properties with Ni-4v/o Y$_2$O$_3$ could be obtained by using the following steps in the processing schedule:

1. Screen prior to and after precleaning.

2. Preclean at 371° C in H$_2$ followed by vacuum treatment.

3. Cold-roll-anneal between 9 and 13 cycles.

Since the highest strength material was the result of processing that involved screening the Ni-4v/o Y$_2$O$_3$ powder, precleaning at 315° C and working 21 cold-roll-anneal cycles, a trade-off could be made for the slightly lower strength (approximately 1.4 MN/m$^2$ (2 ksi)) attained by the above process with the lower number of cold-roll-anneal cycles.

The response of these materials to thermomechanical working does not follow the clearly defined trends exhibited by either the Ni-ThO$_2$ or Ni-Al$_2$O$_3$ material. It would appear from this work that further studies to determine the optimum working conditions for the Ni-Y$_2$O$_3$ material could be of value. This has been borne out in some recent TMP studies conducted at NASA-Lewis wherein the hot rolling temperature and reduction and annealing temperatures have been varied. For example, in these experiments increased hot and cold reductions (percent/pass) appear to yield tensile strengths and rupture lives which are equivalent to or better than data reported here, but with far fewer cold-roll-anneal cycles.

It should further be emphasized that although the 4v/o Y$_2$O$_3$ concentration yielded the highest strength alloy in this study, it has not been determined to be the optimum concentration.
REFERENCES


4. Private communication from Cabot Corp.


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*Calculated ultimate tensile strength.
TABLE I - 1093° C TENSILE TEST DATA FOR NICKEL SHEET DISPERSION
STRENGTHENED WITH 2 AND 4 VOLUME PERCENT YTTRIA

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(MH/m = 315° C (600° F))
Figure 1 - Flow chart for NASCAB process as used in this study.

Figure 2 - Effect of composition on the 1093°C tensile strength of several Ni+Y2O3 alloys.
Figure 3. - Effect of precleaning temperature on the 1093° C tensile strength of Ni4V10Y2O3 alloy.

Figure 4. - 1093° C stress rupture results for Ni4V10Y2O3 alloy.
(a) Ni-4Y₂O₃, TENSILE STRENGTH 187.5 MN/m² (27.2 Ks), SCREENED.

Figure 5. - Comparison of microstructure of Ni-4Y₂O₃ and Ni-2Y₂O₃ composite in the as-rolled condition. Pre-clean temperature, 315° C.

(b) Ni-4Y₂O₃, TENSILE STRENGTH 88.9 MN/m² (12.9 Ks), SCREENED.

Figure 5. - Continued.
(c) Ni-2Y2O3, TENSILE STRENGTH 65.0 MN/m² (9.6 Ks/l), SCREENED.

Figure 5, - Concluded.