(NASA-CR-200037) THE EFFECTS OF INTERSTITIAL CONTENT AND ANNEALING ON THE FLOW AND FRACTURE BEHAVIOR ON POLYCRYSTALLINE BETA-NIAL (NASA. Lewis Research Center) 6 p

N96-17349

Unclas

G3/26 0095137

NASA-CR-200037

NGT-2958

p-0

THE EFFECTS OF INTERSTITIAL CONTENT AND ANNEALING ON THE FLOW AND FRACTURE BEHAVIOR OF POLYCRYSTALLINE β-NiAl

M.L. Weaver*, V. Levit*, M.J. Kaufman* and R.D. Noebe**
*University of Florida, Gainesville, FL 32611-2066
**NASA-Lewis Research Center, Cleveland, OH 44135-3191

IN-26-CR

72285

p-6

ABSTRACT

The strain aging behavior of three polycrystalline NiAl alloys has been investigated at temperatures between 300 and 1200 K. Yield stress plateaus, yield stress transients upon a ten-fold increase in strain rate, work hardening peaks, and dips in the strain rate sensitivity (SRS) have been observed between 700 and 800 K. These observations are indicative of dynamic strain aging (DSA) and are discussed in terms of conventional strain aging theories.

INTRODUCTION

It is commonly accepted that bcc metals, in the presence of sufficient interstitial levels, are subject to the phenomenon of strain aging which can manifest itself as: (1) sharp yield points, (2) serrated stress-strain curves, (3) strain rate sensitivity minima, (4) maxima in plots of work hardening rate as a function of temperature, (5) yield stress plateaus as a function of temperature and (6) flow stress transients upon changes in strain rate (see reference [1]). Not surprisingly, several B2 intermetallic compounds have also been shown to exhibit some of the manifestations associated with strain aging. Despite these observations, the relative significance of strain aging and its influence on the mechanical properties of ordered intermetallic compounds has been largely ignored. The purpose of this document is to describe the results of a study of dynamic strain aging (DSA) in polycrystalline NiAl. To accomplish this goal, stoichiometric NiAl polycrystals with differing interstitial contents were studied. In addition, since dilute additions of reactive elements have been reported to retard strain aging in bcc alloys, an NiAl alloy intentionally doped with Ti was investigated to analyze the role of reactive ternary additions on the strain aging behavior of NiAl.

MATERIALS & METHODS

NiAl in the form of induction melted ingots of (1) conventional purity NiAl (CP-NiAl); (2) carbon-doped NiAl (NiAl-100C); and (3) titanium-doped NiAl (NiAl-Ti) were extruded at 1200 K at an extrusion ratio of 16:1. Post extrusion chemical analyses were conducted using the techniques deemed the most accurate for the particular elements. The results of these analyses are listed in Table 1. Round button-head tensile specimens were ground from the extruded rods so that the gage lengths were parallel to the extrusion direction. Sample dimensions were 3.1 mm for the tensile gage diameters and 30.0 mm for the tensile gage lengths. All specimens were electropolished in a 10% perchloric acid-90% methanol solution that was cooled to 208 K prior to testing.

All tensile tests were performed on an Instron Model 1125 load frame at constant crosshead velocities corresponding to an initial strain rate of 1.4×10^{-4} s^{-1}. Testing was accomplished in three steps. First, baseline room-temperature mechanical properties were determined by testing each alloy as follows: (1) as-extruded and (2) as-extruded + annealed at 1100 K for 7200 s and furnace cooling (FC). The heat treatment temperature was selected based on the prior observations of Margevicius *et al.* [2-5] and Weaver *et al.* [6,7] who showed that sharp yield points could be formed in conventional purity binary NiAl following an annealing treatment of 1100 K/7200 s/FC. Second, the temperature dependence of flow stress

Mat. Res. Soc. Symp. Proc. Vol. 364

was determined by testing all three as-extruded alloys in air between 300 and 1100 K by heating the samples in a clamshell type resistance furnace where temperature gradients were controlled to ±2 K. During this phase of testing, the strain rate sensitivity (SRS) was also determined by increasing the strain rate by a factor of ten from the base strain rate at fixed plastic strain intervals. The quantity extracted from these experiments was the SRS, $s = \Delta\sigma/\Delta\ln\dot{\varepsilon}$. Finally, alloys exhibiting room-temperature yield discontinuities after annealing were subjected to static strain aging (SSA) tests. Description of the SSA test procedure are provided elsewhere [6] .

Table 1. Chemical Compositions of Extruded Alloys Examined in This Study (Atomic Percent)

Alloy	Ni	Al	Ti	C	O	N	S
CP-NiAl	50.1±0.2	49.9±0.2	0.00	0.0186	0.0094	<0.0009	<0.0007
NiAl-100C	49.9±0.2	50.0±0.2	0.00	0.0491	0.0180	<0.0009	<0.0007
NiAl-Ti	49.9±0.2	50.0±0.2	0.03	0.0214	0.0113	<0.0009	<0.0007

Ni & Al Analysis performed using wet chemistry/titration techniques, relative accuracy ±1%.

Ti Analysis performed using Inductively Coupled Plasma Emission Spectroscopy, relative accuracy ±5%

C & S Analysis performed on a Simultaneous Carbon/Sulfur Determinator, LECO Corp., Model CS-244, relative accuracy ±10%

N & O Analysis performed on a simultaneous Nitrogen/Oxygen Determinator, LECO Corp., Model TC-136 or Model TC-436, relative accuracy ±10%

RESULTS

Microstructural Characterization

The post-extrusion microstructures consisted of fully equiaxed and recrystallized grains with nominal grain sizes of 20 μm. The results of chemical analyses of the extruded alloys revealed that within experimental accuracy, the Ni and Al contents of the three alloys are not significantly different from each other. The major differences between the materials are the carbon and oxygen interstitial levels and the addition of 0.025 at.% Ti to NiAl-Ti.

Tensile Properties

The baseline mechanical properties are summarized in Table 2. In agreement with [6], this data showed that the yield stress of each alloy decreased following the 1100 K/7200 s/FC anneal. In addition, a tendency for discontinuous yielding occurred in the CP-NiAl and in NiAl-100C but not in NiAl-Ti after the heat treatment. Analogous yield behavior has been reported previously for conventional purity, cast and extruded or powder processed NiAl alloys [2-6,8-13] and has been attributed to a static strain aging mechanism [6].

Tensile strain rate change experiments were conducted in the temperature range of 300 to 1200 K. The temperature dependent properties (*i.e.*, flow stress, work hardening rate, and SRS) are summarized in Figs. 1-3. Fig. 1 shows the temperature dependence of the flow stress at 0.2% plastic strain. In agreement with previous studies [14], the flow stress decreased gradually with temperature. In CP-NiAl and NiAl-100C, however, an apparent plateau or hump was observed in the range 750-900 K while no such anomalous behavior was apparent for NiAl-Ti.

The work hardening characteristics have been evaluated from the average work hardening rate normalized with respect to elastic modulus $\theta = (\Delta\sigma/\Delta\varepsilon)/E$ (0.2-1.8 percent plastic strain interval) and is summarized in Fig. 2. Much like the 0.2% yield stress, θ decreased steadily with increasing temperature with CP-NiAl and NiAl-100C again exhibiting an anomalous hump in the temperature range 600-700 K. Such a region was not obvious for NiAl-Ti.

Table 2. Baseline Tensile Properties of NiAl Alloys at Room-Temperature

Material	Condition	$\sigma_{0.2}$ (MPa)	σ_f (MPa)	Ductility (%)	Observations
CP-NiAl	as-extruded	163.8	240.5	1.05	CY
	as-extruded	171.6	305.6	1.70	CY
	annealed	116.5	257.3	2.05	DY, sharp YP
	annealed	115.9	335.9	3.34	DY, sharp YP
NiAl-100C	as-extruded	155.1	255.1	1.35	DY, plateau
	as-extruded	151.2	318.8	2.25	DY, plateau
	annealed	112.7	342.1	3.29	DY, sharp YP
	annealed	114.7	285.2	2.35	DY, sharp YP
NiAl-Ti	as-extruded	170.3	235.0	0.86	CY
	as-extruded	176.3	176.3	0.20	CY
	annealed	122.7	255.9	1.76	CY
	annealed	124.2	265.2	1.81	CY

CY = continuous yielding; DY = discontinuous yielding; YP = yield point

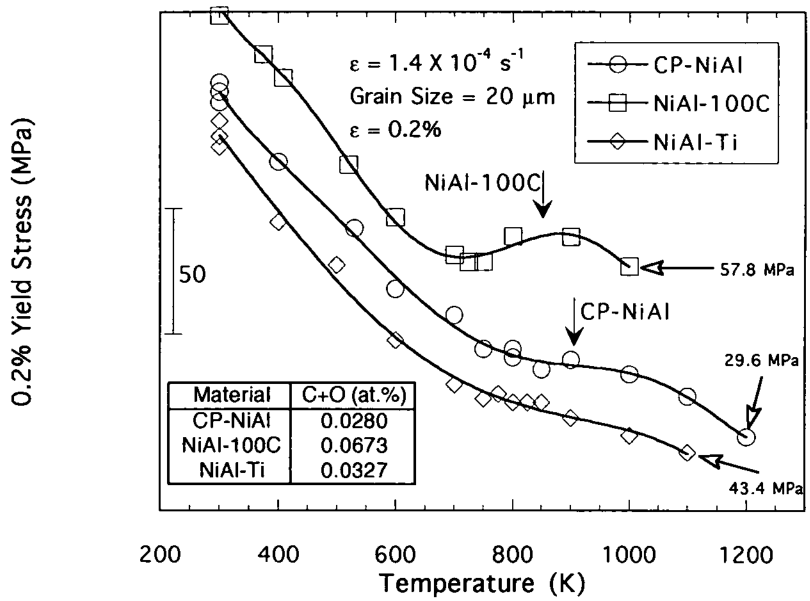

Fig. 1: Temperature dependence of 0.2% yield stress for NiAl Alloys. The solid arrows denote the locations of the flow stress plateaus/peaks.

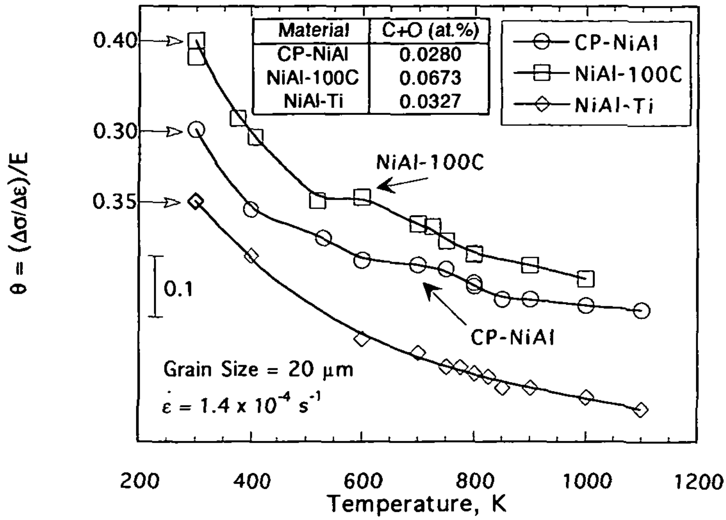

Fig. 2: Temperature dependence of the work hardening rate $\theta=(\Delta\sigma/\Delta\varepsilon)/E$. The solid arrows denote the locations of work hardening peaks.

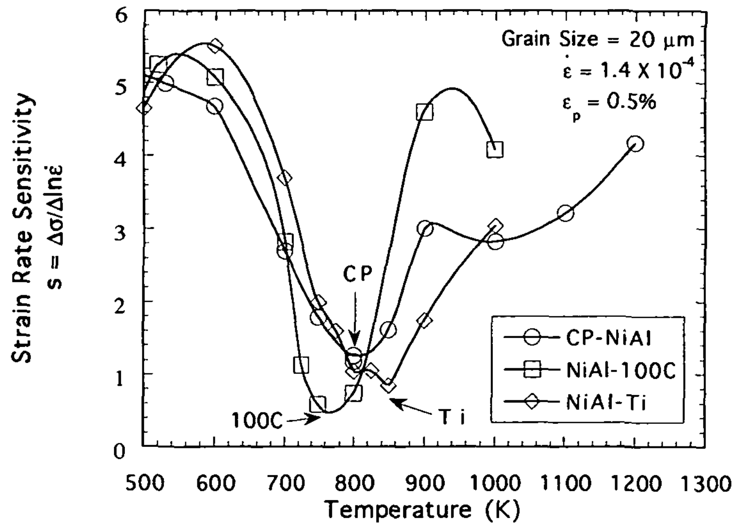

Fig. 3: Temperature dependence of the SRS, $s=\Delta\sigma/\Delta\ln\varepsilon$.

The temperature dependence of the strain rate sensitivity s is presented in Fig. 3. For all three alloys s remained positive but exhibited distinct minima in the temperature range 750-850 K with NiAl-100C occurring first (at 750 K) followed by CP-NiAl and then by NiAl-Ti. This is similar to soft oriented single crystals which also exhibit SRS minima in this regime and which often exhibit negative strain rate sensitivities and serrated flow indicative of DSA [15-

21]. Serrated flow was not observed in any of the alloys studied here, however, yield stress transients in the form of sharp yield points were consistently observed in the temperature range of the flow stress plateau in CP-NiAl and in NiAl-100C upon increasing the strain rate by a factor of 10. This is clearly indicative of DSA. No such yield transients were observed in NiAl-Ti.

Limited SSA experiments were conducted on CP-NiAl and NiAl-100C. These experiments were conclusive in that significant yield points could be obtained for both alloys after aging. For example, in NiAl-100C significant yield points could be recovered after annealing for as little as 2100 s (35 min.) at 622 K which is in perfect agreement with the SSA results of Weaver *et al.* [6].

DISCUSSION

The curves in Figs. 1-3 have several features in common. (1) In CP-NiAl and NiAl-100C, the temperature dependencies of the flow stress and θ increase anomalously in the temperature range 600 to 850 K. Such behavior is not observed in NiAl-Ti. (2) For all three alloys, the SRS's exhibit distinct minima in the temperature range 750-850 K but remain positive. These anomalies are observed at lower temperatures for NiAl-100C followed by CP-NiAl and NiAl-Ti. Similar flow stress peaks and anomalous work hardening parameters have been observed in soft-oriented single crystals in the same temperature range as have strain rate sensitivity minima, negative strain rate sensitivities, and serrated yielding [15-21]. These anomalies are attributed to the migration of interstitial C to dislocations. Confirmation that diffusion of solutes toward dislocations is occurring is provided by the static strain aging studies performed in this study and previously [6,7].

In the present experiments, the flow stress anomalies are shifted to higher temperatures compared with the minimum in SRS. This serves as further evidence in favor of DSA. Classical theory [22,23] dictates that the microscopic mechanism responsible for DSA is the thermally activated motion of dislocations through localized forest obstacles. This type of dislocation motion is characterized by a waiting time, t_w, during which dislocations temporarily arrested at obstacles in the slip path become pinned by diffusing solute atoms. As a result, the obstacles to dislocation motion become stronger with increasing waiting time (*i.e.*, strength increases with increasing temperature and decreasing strain rate) resulting in an increased resistance to plastic deformation. Accordingly, SRS becomes a minimum when the time required to pin a dislocation, t_a becomes equal to t_w. At a given strain rate (*i.e.*, at a fixed t_w) strengthening saturates when the temperature becomes high enough that $t_a \ll t_w$. In other words, maximum strengthening will occur at higher temperatures than SRS minima.

Some interesting observations in NiAl-Ti were: (1) the lack of a flow stress plateau or anomalous work hardening region; (2) the presence of a SRS minima; and (3) the lack flow stress transients upon a change in strain rate. Normally, the temperatures at which DSA phenomena are observed tend to rise with increasing purity. Thus, the temperatures where the flow stress and work hardening anomalies are located should rise with decreasing solute content. Figs. 1-3 show that the stress anomalies and SRS minima do indeed occur at lower temperatures for NiAl-100C compared to CP-NiAl. NiAl-Ti , however, contains just as much C as CP-NiAl. It is therefore suggested that Ti getters some of the C resulting in reduced DSA effects in NiAl-Ti. Further experiments are in progress to confirm this hypothesis.

SUMMARY AND CONCLUSIONS

1. The three alloys investigated in the present study exhibit pronounced minimas in SRS in the temperature range 750 to 850 K which are indicative of DSA.

2. The occurrence of SSA [6,7], yield points during room temperature testing after annealing, yield stress transients upon changes in strain rate , and the occurrence of yield stress and work hardening anomalies confirm that DSA does occur in polycrystalline NiAl alloys. Serrated yielding was not observed in polycrystalline NiAl.

3. Preliminary analysis indicates that the addition of dilute quantities of reactive metals can reduce the effects of DSA via a gettering mechanism. Certainly this aspect deserves more study.

ACKNOWLEDGMENTS

Helpful discussions with A.J. Duncan (UF), J.M. Brzeski (Yale), J.E. Hack (Yale) and R.E. Reed-Hill (UF) are gratefully acknowledged. MLW would like to thank the NASA-Lewis Research Center for financial support of this research under Grant NGT-2958.

REFERENCES

1. P. Rodriquez, *Bull. Mat. Sci.* **6**, 653 (1984).
2. R. W. Margevicius and J. J. Lewandowski, *Acta Metall. Mater.* **41**, 485 (1993).
3. R. W. Margevicius and J. J. Lewandowski, *Scripta Metall. Mater.* **25**, 2017 (1991).
4. R. W. Margevicius, J. J. Lewandowski, I. E. Locci and R. D. Noebe, *Scripta Metall. Mater.* **29**, 1309 (1993).
5. R. W. Margevicius, J. J. Lewandowski and I. E. Locci, *Structural Intermetallics*, R. Darolia, J. J. Lewandowski, C. T. Liu, P. L. Martin, D. B. Miracle and M. V. Nathal, Eds., The Minerals, Metals and Materials Society, Seven Springs, PA, p. 577 (1993).
6. M. L. Weaver, R. D. Noebe, J. J. Lewandowski, B. F. Oliver and M. J. Kaufman, *Mater. Sci. Eng.* , in press (1994).
7. M. L. Weaver, M. J. Kaufman and R. D. Noebe, *submitted to Acta. Metall. Mater.* , (1994).
8. R. D. Noebe and A. Garg, *Scripta Metall. Mater.* **30**, 815 (1994).
9. R. D. Noebe and M. K. Behbehani, *Scripta Metall. Mater.* **27**, 1795 (1992).
10. A. G. Rozner and R. J. Wasilewski, *J. Inst. Metals* **94**, 169 (1966).
11. K. H. Hahn and K. Vedula, *Scripta Metall.* **23**, 7 (1989).
12. M. Dollar, S. Dymek, S. J. Hwang and P. Nash, *Metall. Trans. A* **24A**, 1993 (1993).
13. R. T. Pascoe and C. W. A. Newey, *Metal. Sci. J.* **2**, 138 (1968).
14. R. D. Noebe, R. R. Bowman and M. V. Nathal, *Int. Mater. Rev.* **38**, 193 (1993).
15. M. L. Weaver, unpublished research, University of Florida (1994).
16. J. E. Hack, J. M. Brzeski and R. Darolia, *Scripta Metall. Mater.* **27**, 1259 (1992).
17. J. E. Hack, J. M. Brzeski, R. Darolia and R. D. Field, *High-Temperature Ordered Intermetallics V*, I. Baker, R. Darolia, J. D. Whittenberger and M. H. Yoo, Eds., Materials Research Society, Boston, MA, p. 1197 (1993).
18. J. E. Hack, J. M. Brzeski and R. Darolia, *Mater. Sci. Eng.* , in press (1994).
19. K. Kitano and T. M. Pollock, *Structural Intermetallics*, R. Darolia, J. J. Lewandowski, C. T. Liu, P. L. Martin, D. B. Miracle and M. V. Nathal, Eds., The Minerals, Metals and Materials Society, Seven Springs, PA, p. 591 (1993).
20. K. Kitano, T. M. Pollock and R. D. Noebe, *Scripta Metall. Mater.* **31**, 397 (1994).
21. J. M. Brzeski, J. E. Hack, R. Darolia and R. D. Field, *Mater. Sci. Eng.* **A170**, 11 (1993).
22. L. P. Kubin and Y. Estrin, *J. Phys. III* **1**, 929 (1991).
23. P. G. McCormick, *Acta Metall.* **20**, 351 (1972).