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**EFFECT OF Al CONTENT ON MICROSTRUCTURE/
PHASE DISTRIBUTION AND STRENGTH/DUCTILITY IN
A PM GAMMA ALLOY (PREPRINT)**

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Metals Branch

Metals, Ceramics, and NDE Division

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Effect of Al Content on Microstructure/Phase Distribution and Strength/Ductility in a PM Gamma Alloy

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ABSTRACT

The strength and ductility variation of TiAl alloys with variations of microstructure and composition are quite complicated and, possible interactions between these effects exist. For example, Al is known to affect strength and the variation in volume fraction of gamma and alpha-2 phases have been credited with the effect. However, the impact of composition versus phase fraction and microconstituent fraction has not been satisfactorily quantified, especially for multi-element compositions. In an effort to understand such effects, four billets of gamma alloy 03K, nominal composition of Ti-45.5Al-3Nb-1Cr-0.2W-0.2B-0.4C-0.2Si (at%), were produced from gas-atomized powder. The actual Al content varied between 44.8 and 47.4 at%. The forgings were heat treated to develop three microstructural variants: fully lamellar, nearly lamellar and duplex. The microstructure and Rockwell C hardness were analyzed on all samples, and room-temperature tensile testing was conducted on the fully-lamellar materials. The resulting strength and ductility as a function of Al content and microstructure will be discussed.

INTRODUCTION

The composition-microstructure-property relationships in TiAl-based alloys are quite complex and have been under investigation since the ground breaking work of Blackburn,¹ who first demonstrated the effect of aluminum content on properties of binary gamma titanium-aluminide alloys. Over the course of the following two decades several studies (this list is by no means all inclusive) clearly articulated the sensitivity of mechanical properties to alloying additions, with strength and ductility being mostly sensitive to aluminum content and grain size.^{2,3,4,5} The general trend is marked by increased ductility and decreased strength with increasing aluminum content for two-phase alloys. When the Al content is sufficiently high, that is greater than 50% in most binary and ternary alloys studied, the material consists of singularly gamma phase and the ductility drops precipitously. The results of Huang concluded that the plastic fracture strain peaked at Al concentrations between 46 and 48 at% for the binary Ti-xAl, and many ternary Ti-xAl-yM, alloys where yM was 2Cr, 3V, or 2Mn for alloys processed via investment casting.³ Huang also demonstrated that in cast alloys this range of Al concentration (not coincidentally) resulted in a fracture toughness, K_Q , peak and, that K_Q dropped dramatically for the single-phase alloys (Al concentrations >50 at%). Similarly, Austin and Kelly showed a ductility peak at an Al concentration between 47 and 48 at% in the Ti-xAl-2Cr-2Nb quaternary system.⁵ However, the increased ductility came at the expense of strength, with the low ductility/low Al alloys possessing yield strengths as much as 67 percent higher, 500 versus 300 MPa.

Confounding the observations of Al-concentration effects on the properties of two-phase alloys is the fact that microstructure can, and often did, vary as a function of composition. Low-Al alloys tended to be lamellar and high-Al alloys often exhibited a duplex or near-gamma microstructure when processed by casting and heat treatment. Kim demonstrated, using several multi-component alloys, Ti-47Al-1Cr-1V-2.5Nb and Ti-46.5Al-2.1Cr-3.0Nb-0.2W to list two, that multiple microstructure variations could be achieved for a given composition. He outlined the heat treatment schemes to achieve fully lamellar (FL), nearly lamellar (NL), duplex (DP) and near-gamma (NG) microstructures.⁶ He also demonstrated the effect that these microstructure variations had on strength, ductility and toughness.^{6,7} In general, NG and DP microstructures provided the greatest ductility, but yielded the lowest fracture toughness, while FL microstructures had poor ductility, but twice the fracture toughness. However, Kim also observed that the strength of the FL materials was a strong function of grain size, with a finer grain size (<500 μm) resulting in strengths that were approximately 40% greater than large-grain material (>1000 μm), 500 versus ~350 MPa, respectively. The increased strength came with a minor impact on fracture toughness, K_{IC} of 22.8 versus 26-32 $\text{MPa}\sqrt{\text{m}}$. Noteworthy in those studies was that the NL microstructure produced seemingly the best balance of strength and ductility, but fracture toughness was not evaluated. Subsequently, Dimiduk et al. concluded that lamellar spacing was more significant than grain size in the strengthening of FL TiAl alloys.⁸ In this work lamellar spacing and grain size could not be completely decoupled. The grain size, D , ranged from 56 to 406 μm , and the average lamellar spacing, λ , ranged from 35 to 149 nm with a general observation of finer lamellar spacing associated with smaller grain size. Kim also acknowledged the role of lamellar spacing in the Hall-Petch strengthening relationship for FL materials in his subsequent research.⁹ He also noted that ductility was directly related to grain size, with increased ductility with decreasing grain size. These later studies concluded that the preferred path to achieve the desired properties in wrought TiAl-based materials was through control of both the FL grain size and lamellar spacing.

Unfortunately, what is required from an application standpoint is a balance of properties: strength, ductility, toughness, creep, oxidation resistance, and fatigue resistance to name some key properties of envisaged turbine engine components. Thus, the alloy development community has been on a quest for a composition/microstructure that possesses the attractive strengths of some of the early lower-Al compositions, but retains adequate ductility and toughness.^{10,11,12,13} Recent development work investigating “third generation” gamma TiAl alloys, reported on by Stanley, formulated the material used in this study. The alloy, referred to as 03K, has a nominal composition of Ti-45.5Al-3Nb-1Cr-0.2W-0.2B-0.4C-0.2Si (at%), and was selected “... based on it exhibiting a better balance of properties, particularly preserving some room temperature ductility.”¹³ Subsequently, difficulties were encountered in forging billets of the powder-metal based material.¹⁴ The uncertainty in the Al content and the availability of small amounts of remnant powder from individual powder runs led to this investigation of the sensitivity of 03K to unintended variations in Al content.

EXPERIMENTAL PROCEDURES

Material for this research was provided by Crucible Research in the form of four billets. Each billet had been formed by hot isostatically pressing remnant powder from individual powder runs. The HIP parameters were 1204°C/104MPa/3h, and the billets were nominally 100 mm in diameter and 150 mm in length. Chemical analyses conducted on the alloy powders showed that the four batches had significantly different aluminum contents, ranging from 44.80 to 47.40

(at%). For ease of discussion the four materials will be referred to as 3KA, 3KB, 3KC, and 3KD, as listed in Table 1, and in increasing order of Al content.

Table 1: Selected 03K Alloy Powder Heats and Analyzed Compositions (at%)

<i>ID</i>	<i>Al</i>	<i>Cr</i>	<i>Nb</i>	<i>W</i>	<i>B</i>	<i>Si</i>	<i>C</i>	<i>O (wt%)</i>
3KA	44.80	0.94	2.98	0.17	0.190	0.210	0.460	0.074
3KB	45.30	1.00	3.11	0.23	0.220	0.210	0.450	0.058
3KC	46.80	0.97	2.97	0.18	0.210	0.180	0.360	0.078
3KD	47.40	0.94	2.95	0.17	0.210	0.180	0.380	0.074

Each billet was forged at after soaking at a temperature of 30-45°C below the alpha-transus temperature, T_α . Since T_α has been demonstrated to be a strong function of Al concentration,³ it was first necessary to establish T_α of each billet. This was accomplished extracting a small sample from each billet and conducting differential thermal analysis (DTA). DTA consisted of two heating-cooling cycles. First the sample was heated from ambient temperature to 1450 or 1500°C and then cooled to 1000°C, all at a rate of 20°C/min. The sample was then heated back to 1400°C, and then cooled to room temperature. The alpha transus temperature for each composition was determined by analyzing the local minimum of the endothermic peaks during the second heating cycle. As an example the DTA for 3KB is provided in Figure 1. T_α of each billet is provided in Table 2.

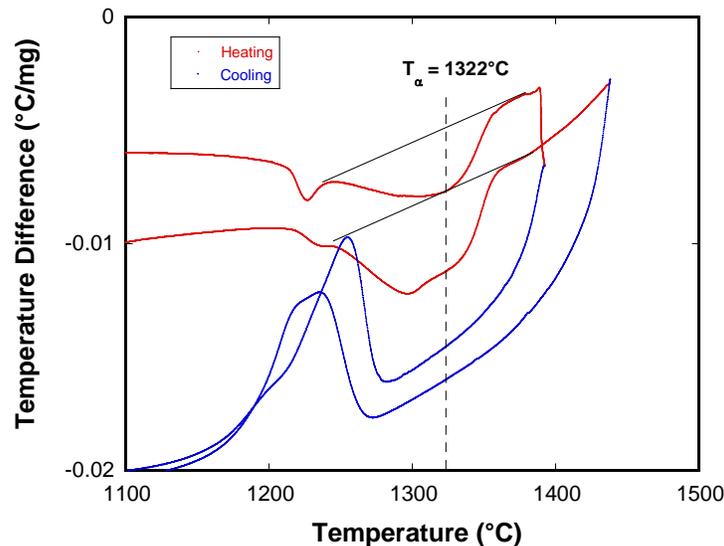


Figure 1. Determination of T_α for 3KB using differential thermal analysis.

Prior to forging, 76 mm diameter x 102 mm long forging preforms were EDM machined from each billet. The preforms were coated with CaO, wrapped with high temperature ceramic cloth, and then canned in a 0 mm thick stainless steel can having an air exit hole on the wall. During the course of preform preparation, the 3KA material developed surface cracking, which required further machining to remove the cracked material. As a result the final forging preform for 3KA was smaller, approximately 55 mm diameter x 89 mm long. The canned preform was soaked in

air at a temperature of 30-40°C below the respective T_{α} , where a stable, fine-grained duplex microstructure could form. The preform was then transferred on to open flat dies heated at 370°C, and forged to an approximately 80% reduction under an initial forging rate of $\sim 5 \times 10^{-2}/s$. The dwell time from the furnace to the onset of deformation was 35sec for all forging and the forged pancake was set aside to cool in air on a brick. The pancakes were de-canned by circumferential cutting with water-jet and lifting up the can layers from both sides of the pancakes. The resulting pancakes had dog-bone shaped cross-sections when diagonally sectioned, with the mid section significantly thinner than the edges, as shown in Figure 2 and indicated in the last column of Table 2.

Table 2: Forging Conditions and De-canned Pancake Shape/Dimensions

Alloy ID	T_{α} (°C)	Preform Size D x L	Soaking (°C/h)	Pancake Size D x t (cm)
3KA	1297	5.5 x 8.9	1270/2	11 x (2-0.6)
3KB	1322	7.6 x 10.2	1290/2	15 x (3-1)
3KC	1342	7.6 x 10.2	1310/1.5	15 x (3-1)
3KD	1361	7.6 x 10.2	1320/1.5	15 x (3-1)

D (diameter); L (length); t (thickness)



Figure 2: An 03K pancake segment showing the dog-bone shape cross-section

Heat treat (annealing) experiments were conducted on samples from the forged material at temperatures above (by $\sim 20^\circ\text{C}$) and below (by $\sim 40^\circ\text{C}$) the respective T_{α} , followed by different cooling schemes. Microstructure evolution was investigated via back-scattered electron imaging (BEI) and analysis of phase volume distributions after water quenching as well as following controlled cooling (furnace cooling to 900°C and then air cooling). Based on experience from previous microstructure evolution studies, heat treatments were designed to generate a composition-specific fully lamellar microstructure, Table 3. Resulting microstructures were investigated using BEI and the distributions of constituent phases were analyzed on selected microstructure forms using orientation imaging microscopy (OIM).

Two dog-bone tensile specimens were machined from each of the heat-treated pancakes. Nominal specimen dimensions are provided in Figure 3. Note that the specimens from 3KA were undersized relative to the other three compositions tested. This was necessitated by the reduced physical size of the 3KA pancake. Tension tests were conducted at room temperature at a strain rate of $4 \times 10^{-4}/s$. Due to uneven and different pancake thicknesses and different diameters, their configurations and dimensions had to be designed under various constraints.

Nevertheless, the thinnest gage diameter (1.6 mm or 1600 μm) of the 3KA specimens should contain at least 10 grains along the diagonal. Rockwell “C” hardness measurements were conducted on various microstructure forms in order to correlate with the corresponding tensile strengths and project tensile strengths for the microstructures that were not tested.

Table 3: Alpha-anneal heat treat conditions and microstructure for tensile specimens.

ID	Heat Treatment Condition	Microstructure	Phase volume fraction		
			γ	α_2	β
3KA	1325°C/1.5h/FC/900°C/18h/AC	Fully-Lamellar	90.9	7.9	1.2
3KB	1345°C/1.5h/FC/900°C/18h/AC	Fully-Lamellar	95.6	3.8	0.6
3KC	1370°C/1.2h/FC/900°C/18h/AC	Fully-Lamellar	95.4	3.7	0.9
3KD	1388°C/1h/FC/900°C/18h/AC	Degenerated FL	96.8	2.4	0.8

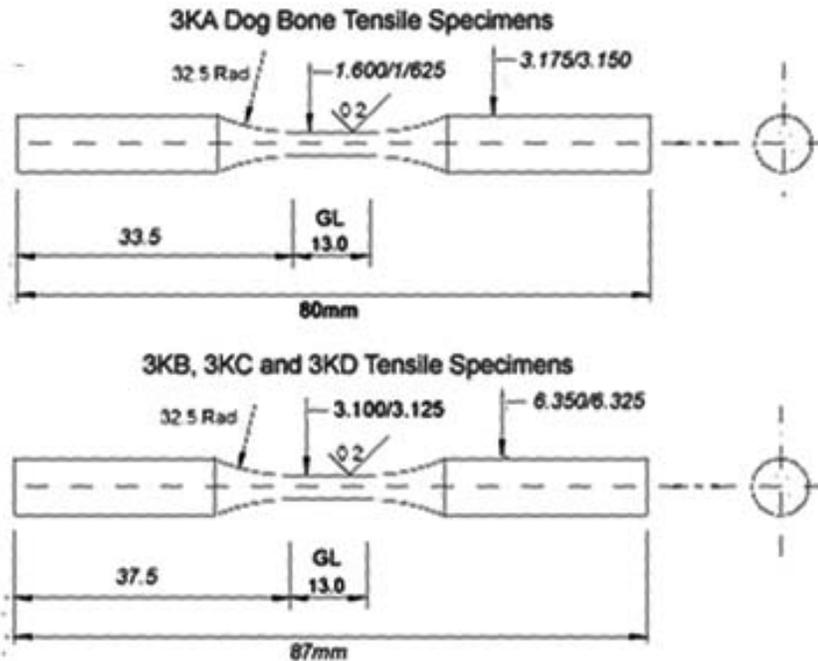


Figure 3. Dog-bone tensile specimens utilized.

RESULTS and DISCUSSION

The alpha heat treatment, consisting of annealing in the alpha field and subsequent controlled cooling, yielded fully lamellar microstructures for 3KA, 3KB and 3KC, exemplified in Figure 4 for 3KA and 3KC and Figure 5a for 3KB. Qualitatively, the grain sizes were roughly comparable, in the neighborhood of 100 – 200 μm . However, the grain boundary morphologies differed in that increased aluminum content led to more tortuous or rugged boundaries, Figure 4a and 4c. Noteworthy is that the lamellar spacing increased with increased aluminum content, i.e. finer lamellae for lower Al concentration, which is clear from comparing 3KA and 3KC, Figure 4b and 4d, respectively. Recall that Dimiduk et al. concluded that lamellar spacing dominates yield strength irrespective of grain size.⁸ This would suggest that tensile properties should scale with Al content as a result of the lamellar structure realized in the alpha annealing heat treatments provided. The lamellar microstructure realized in alloy 3KD (highest Al content),

Figure 5c, differed in morphology from the other compositions. This so called ‘degenerated lamellar structures’ has previously been observed in gamma alloys containing relatively high Al content, and the formation mechanism is not well understood.⁶ When annealed at 40°C below T_{α} , typical duplex microstructures having fairly uniform and fine grain sizes resulted, as shown in Figure 5 where both FL and duplex microstructures are compared for alloys 3KB and 3KD. The average grain sizes of the duplex microstructures ranged from 6.8 to 7.7 μm . The phase distribution of the FL samples was further analyzed by OIM to quantify the phase volume fractions. Only a small confined area (400 x 400 μm) was evaluated, but it validated the general trend that the α_2 / gamma ratio increases with decreasing aluminum content. The results of the phase distribution determined for each alloy sample is listed in Table 3.

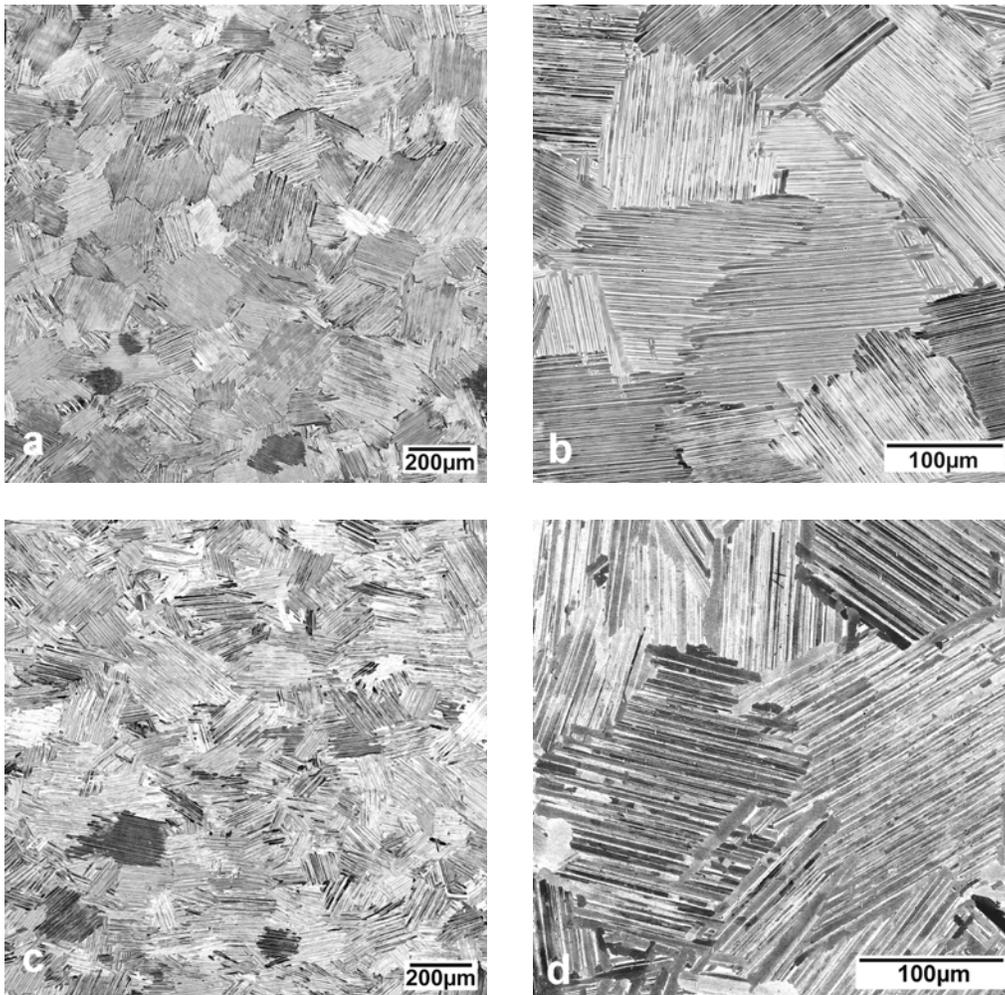


Figure 4: Fully-lamellar microstructures at two different magnifications in 3KA (a, b) and 3KC (c, d) samples generated by the alpha treatment followed by controlled cooling.

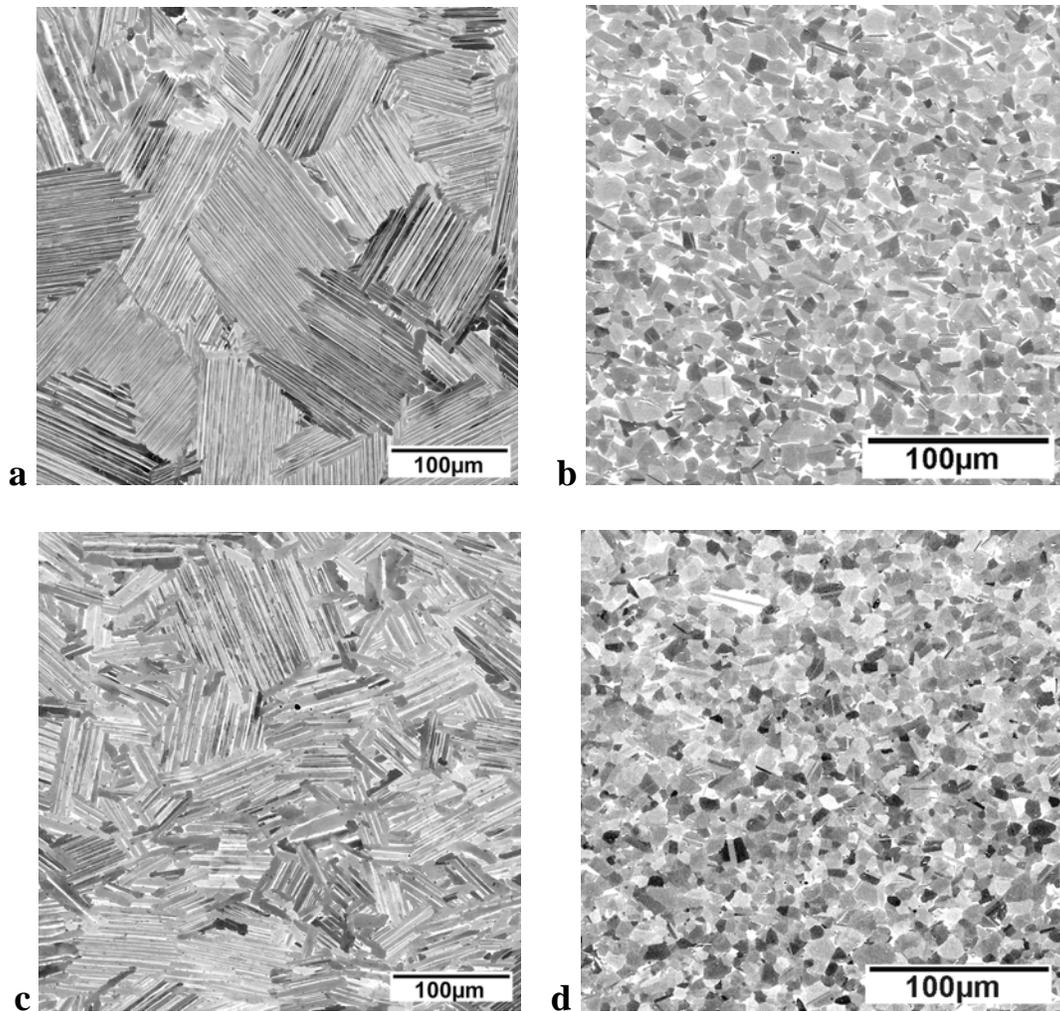


Figure 5: Microstructures of 3KB and 3KD in both fully-lamellar and fine duplex: 3KB fully-lamellar (a), 3KB fine duplex (b), 3KD fully-lamellar (c), and 3KD fine duplex (d).

Two room temperature tension tests were performed on each composition after alpha-anneal heat treatment to achieve the FL microstructures discussed previously. Representative tensile curves are displayed in Figure 6. Noteworthy was that the ductility of the 3KA material (44.8 at% Al, the highest α_2 volume fraction, and finest lamellar spacing) exhibited negligible plastic strain. The 3KB specimen just reached the 0.2% offset, with a yield strength of 607 MPa. The strain-to-failure was 1% and 2% greater for the 3KC and 3KD specimens, Al concentrations of 46.8 and 47.4 at%, respectively. Accompanying the increased ductility was a decrease in yield strength to approximately 500 and 460 MPa. Also apparent was a change in initial hardening rate, just after the proportional limit (onset of yielding). The hardening rate of the 3KD specimen achieved the long term hardening rate immediately at the onset of plastic flow, and the stronger, less ductile, specimens exhibited a decreasing hardening rate with increasing strain until at least 0.5% strain had been achieved. Investigation into the hardening mechanisms and subsequent degradation of ductility was beyond the scope of this investigation; however, it is likely a result of the decreased volume fraction of α_2 and increased lamellar spacing. Irrespective of insight into the actual mechanisms operating, one must be concerned, from a

ductility standpoint, of Al concentrations less than about 46 at% for the 03K composition. The cliff in ductility could certainly impact processing, handling, and durability.

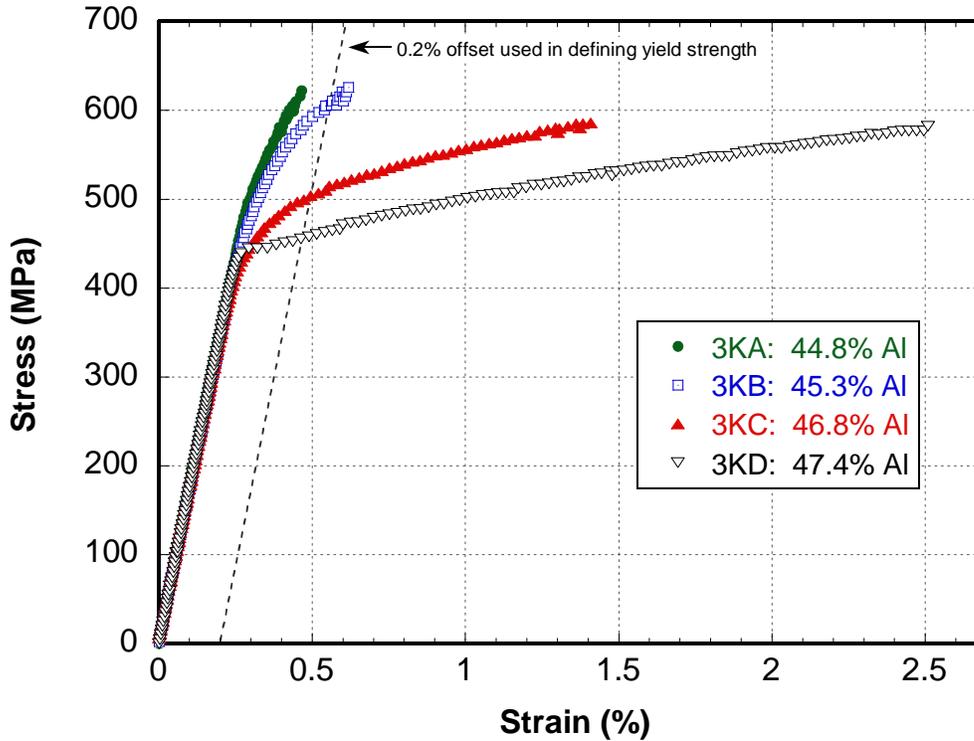


Figure 6. Tensile curves of fully-lamellar 03K as a function of Al concentration.

Rockwell hardness, R_c , was measured on samples of all three microstructure variants (fully-lamellar, nearly-lamellar, and fine duplex) of each composition. The results are provided in Table 4, where each value represents the average of six measurements. Subtle variations in hardness were realized for a given chemistry, with the nearly lamellar (NL) always having the lowest hardness. Dimiduk demonstrated that 0.2% yield stress trended with R_c for FL microstructures of Ti-45.3Al-2.1Cr-2.0Nb.⁸ Kim¹⁵ added to that data set with results for FL microstructures of two additional alloys, K7 and K5. Details of the K7 and K5 alloys can be found in reference [7]. Results from those alloys are depicted in Figure 7 along with a trend fit to the data. Test results the FL 03K material (column 2 in Table 4 and the strengths from Figure 6) are included for comparison. They, too, are consistent with the observed exponential trend. This suggests that Rockwell hardness can be an effective screening test for strength.

Table 4. Rockwell “C” hardness results.

Composition ID	Rockwell “C” Hardness		
	Fully Lamellar (FL)	Nearly Lamellar (NL)	Fine Duplex (FD)
3KA	35.0	33.9	34.8
3KB	34.3	32.7	33.9
3KC	31.0	29.7	32.7
3KD	28.2	27.5	30.4

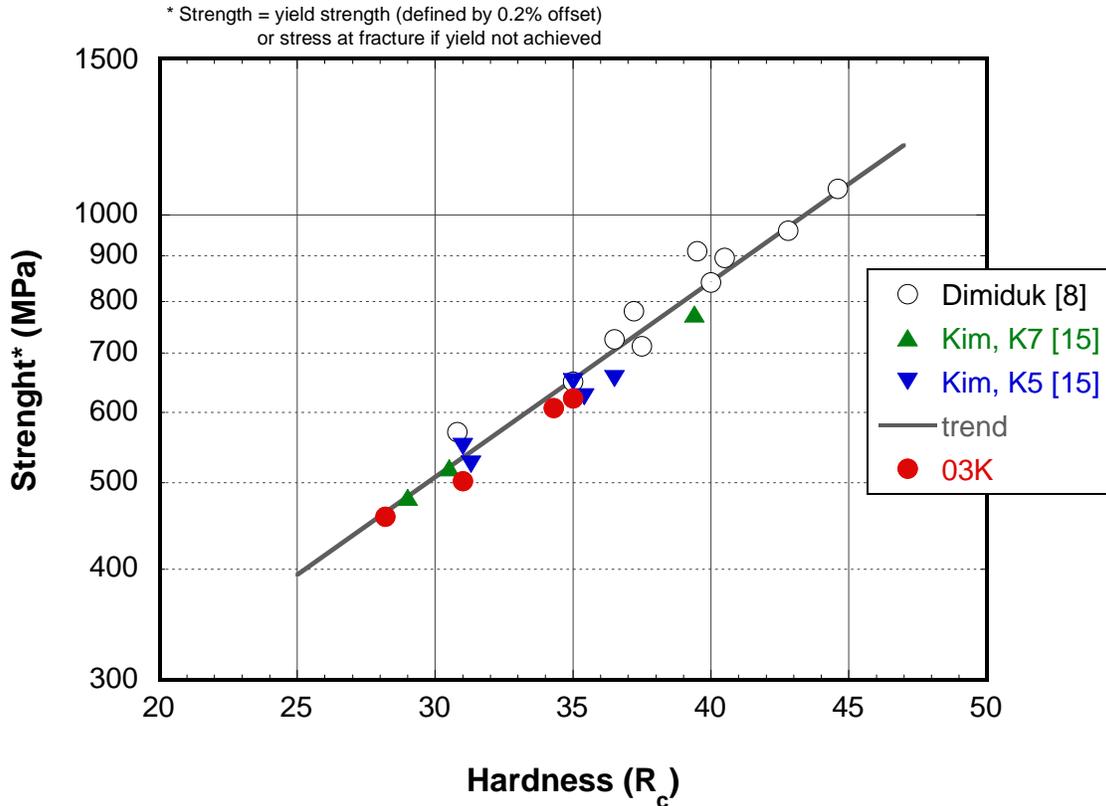


Figure 7. Trend of yield strength versus hardness for FL microstructures of various TiAl alloys.

If one assumes the trend observed for FL microstructures also applies to the other microstructures realized, then the hardness results in Table 4 of most interest are those for 3KC in the fine-duplex structure. The fine-duplex microstructure is expected to have improved ductility, at the expense of fracture toughness, with a strength approaching 600 MPa, if the trend holds. This is one possible path to a higher strength TiAl material with acceptable room temperature ductility, but it has yet to be proven.

SUMMARY and CONCLUSIONS

Four pieces of a multi-element gamma titanium aluminide alloy, Ti-45.5Al-3Nb-1Cr-0.2W-0.2B-0.4C-0.2Si (at%), were examined investigating the sensitivity to slight variations from the target chemistry, paying particular attention to variations in Al concentration. Test materials were machined from pancakes forged high in the alpha-gamma phase field and heat treated to achieve fully lamellar, nearly lamellar, and duplex microstructures. Room temperature tensile properties were measured on the FL microstructures and Rockwell C hardness was analyzed on all samples. The Al content of the starting powder lots varied from 44.8 to 47.4 at%.

Trends were as expected with increasing Al concentration and included: increased alpha transus temperature, decreased α_2 volume fraction, increased lamellar spacing and grain boundary tortuosity. For the fully lamellar material increasing Al concentration resulted in decreased yield strength, and increased ductility or strain-to-failure. Noteworthy were the lack of ductility for

the specimens with ≤ 45.3 at% Al and the change in initial work hardening rate. The onset of plastic flow was not significantly affected by the Al concentration.

Rockwell hardness results showed a minimum for the nearly lamellar microstructure for all four compositions. A trend of yield stress with hardness was proposed and the FL 03K materials tested were consistent with the observed trend developed for other TiAl alloys. If this trend holds for other microstructures, it suggests that the fine duplex microstructure for compositions with Al concentration > 46 at % may have a balance of strength and ductility. However, previous studies have shown that this may come at the expense of fracture toughness.

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