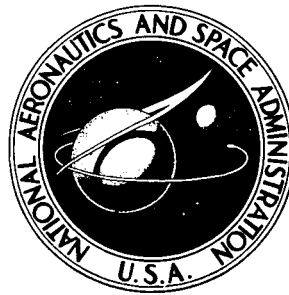


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**EXPLORATORY STUDY OF ELEVATED-TEMPERATURE
TENSILE PROPERTIES OF ALLOYS BASED ON
THE INTERMETALLIC COMPOUND TiNi**

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16. Abstract The tensile properties and oxygen contamination behavior of TiNi alloyed with aluminum, chromium, and silicon were investigated in the temperature range between 800 and 1000 K (980° and 1340° F). The alloys were significantly stronger than unalloyed TiNi and less susceptible to embrittlement than the Ti-6242 alloy.			
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EXPLORATORY STUDY OF ELEVATED-TEMPERATURE TENSILE PROPERTIES OF ALLOYS BASED ON THE INTERMETALLIC COMPOUND TiNi

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SUMMARY

The tensile properties and oxygen contamination behavior of the equiatomic titanium-nickel intermetallic compound (TiNi) and several of its alloys were examined in the temperature range between 800 and 1000 K (980⁰ and 1340⁰ F). Additions of aluminum, chromium, and silicon increased the elevated-temperature tensile strength of TiNi as much as threefold. Both TiNi and its alloys are less susceptible to elevated-temperature oxygen contamination than is the Ti-6242 alloy. The elevated-temperature tensile strength of TiNi is not affected by its 900 K (1160⁰ F) allotropic transformation. Aluminum and possibly chromium promote solid-solution softening in TiNi at low solute concentrations.

INTRODUCTION

The upper temperature for the useful application of commercial titanium alloys in turbine engines is approximately 750 K (890⁰ F). The most recent experimental alloys have raised this temperature limit to somewhat above 800 K (980⁰ F); but considered as a whole, the various strength properties of titanium alloys begin to degrade rapidly above 750 K (890⁰ F). Below this temperature the strength properties generally decrease only moderately with increasing temperature.

The effect of this limitation can be seen in the materials considered for the blades of the various compressor stages of a typical advanced turbine engine. These blades are subjected to temperatures between approximately 500 and 900 K (440⁰ and 1160⁰ F), as shown in figure 1. Through stage 6, near 750 K (890⁰ F), the titanium alloy selected has useful creep strength. Above this temperature, however, a nickel-base superalloy must be used. The creep strength of the nickel alloy greatly exceeds that of the titanium alloy. In fact, the strength is so much greater that even when normalized with respect to specific gravity as shown in figure 2, at 850 K (1070⁰ F) the nickel alloy has four times the

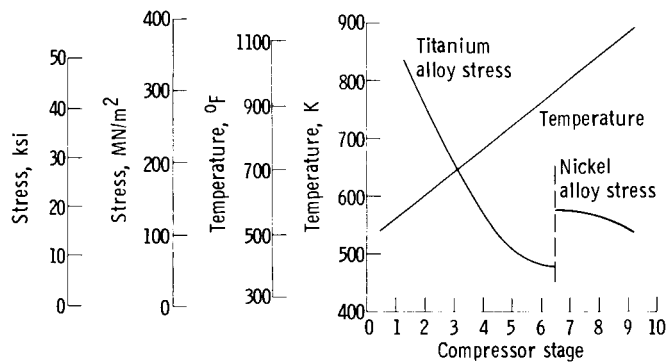


Figure 1. - Blade temperature, stress, and composition at various compressor stages for a typical advanced turbine engine under cruise flight conditions.

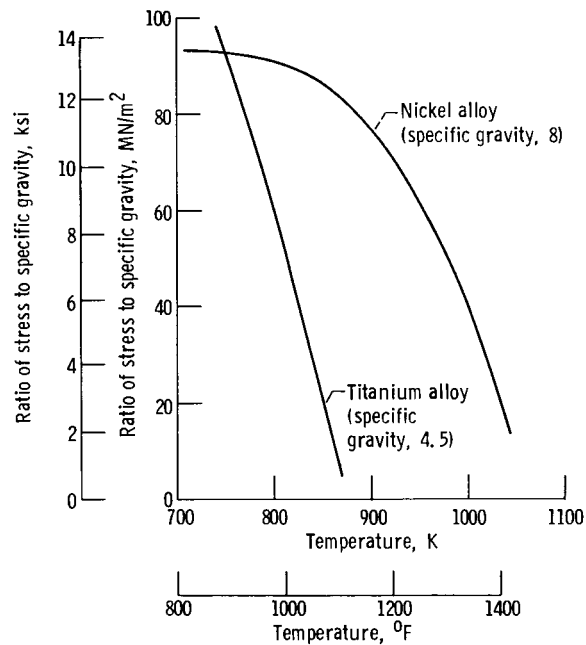


Figure 2. - Specific-gravity-compensated stress required to produce a design creep strain in 100 hours in titanium and nickel alloys considered for advanced turbine engines.

creep strength of the titanium alloy. Unfortunately, the nickel alloy has a specific gravity almost twice that of the titanium alloy (8, as compared to 4.5), which results in both a greater blade stress due to centrifugal loading and a significantly higher engine weight.

Thus, though it entails a weight penalty, the nickel-base alloys must be used in the latter stages of the compressor because of the inadequate strength of titanium alloys. Moreover, the use of titanium alloys is further complicated by the susceptibility of titanium to oxygen contamination above approximately 850 K (1070° F).

In light of these considerations, a different type of possible blade material for the latter stages of a compressor was studied, an intermetallic compound. Only one intermetallic compound with useful ductility has been studied in any detail, the equiatomic compound titanium-nickel (TiNi). It has a specific gravity of 6.45, intermediate between titanium and nickel. The compound TiNi has been studied in detail by many investigators primarily because of its unique memory properties, which are associated with a low-temperature martensitic transformation, with the M_s temperature just below 450 K (350° F). Consequently, few high-temperature tensile property determinations have been made.

This study was conducted to survey the tensile properties and oxygen contamination behavior of TiNi and several of its alloys in the temperature range between 800 and 1000 K (980° and 1340° F), a range of temperatures above that in which current titanium alloys are useful as compressor blade materials. These properties are compared with those of the advanced titanium alloy Ti-6242 (Ti-6Al-2Sn-4Zr-2Mo).

MATERIALS AND PROCEDURE

Materials and Specimen Preparation

Equal atomic amounts of titanium and nickel with the alloying additions listed in table I were triple arc-melted under argon into 60-gram (2.1-oz) buttons. These alloys were then drop cast into cylinders approximately 15 millimeters (0.59 in.) in diameter and 30 millimeters (1.2 in.) in length. In addition, annealed Ti-6242 rods were machined to these dimensions. These materials were steel jacketed and extruded at 1200 K (1700° F) at a reduction ratio of 8:1. The steel jackets were removed by acid leaching and the extrusions swaged at 1100 K (1520° F) to 3 millimeter (0.12-in.) rods. The rods were sectioned and centerless ground to the tensile specimen configuration shown in figure 3.

The concentrations of titanium and nickel in the alloys investigated were within 1 atom percent of each other.

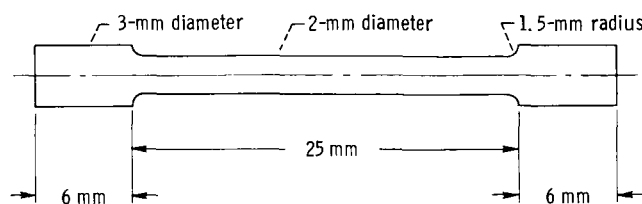


Figure 3. - Buttonhead tensile specimen configuration for TiNi alloys and Ti-6242.

Experimental Procedure

The tensile specimens were tested at room temperature and at 800, 850, 900, 950, and 1000 K (980^o, 1070^o, 1160^o, 1250^o, and 1340^o F) at a crosshead speed of 0.020 millimeter per second (0.047 in./min). The specimens were tested in the as-swaged condition unless subjected to a prior oxidation exposure. The Ti-6242 was also tested in the as-swaged condition. The measured strength of Ti-6242 is not less than the published values for duplex annealed material (ref. 1).

Tensile specimens of several of the alloys investigated were oxidized in still air in silica boats in an open vertical tube furnace for various lengths of time at 900 and 1000 K (1160^o and 1340^o F). Their ductilities as compared to unexposed specimens are employed as a measure of susceptibility to embrittlement, with the assumption that loss of ductility can be attributed solely to oxygen contamination.

Considerable difficulty was encountered in the metallographic preparation of the alloys. The common etchants suitable for titanium alloys (33CH₃COOH-33HNO₃-33H₂O-1HF) and TiNi (82H₂O-14HNO₃-4HF) simply pitted the TiNi alloys. Only the nickel etchant 30HN₄C1-50HNO₃-20H₂O was able to partially delineate grain structures. The specimens were optically examined at 250X to determine their structural characteristics. The specific gravities of the alloys examined were determined by the water displacement method.

RESULTS

The ratio of ultimate stress to specific gravity of TiNi at elevated temperatures is shown in figure 4 (table II), where it is compared with that of the advanced titanium alloy, Ti-6242. The intermetallic compound was considerably weaker than the titanium alloy, as would be expected, because TiNi is a stoichiometric compound without benefit of strengthening additions.

Additions of as much as 3.4 atomic percent aluminum were examined in this study. Above this concentration of aluminum the extrusions could not be swaged. At low con-

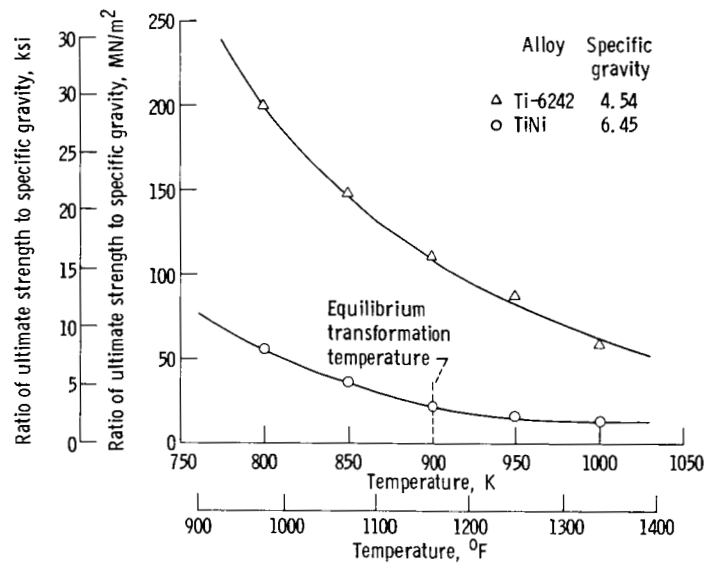


Figure 4. - Specific-gravity-compensated strength of TiNi and Ti-6242 at elevated temperatures.

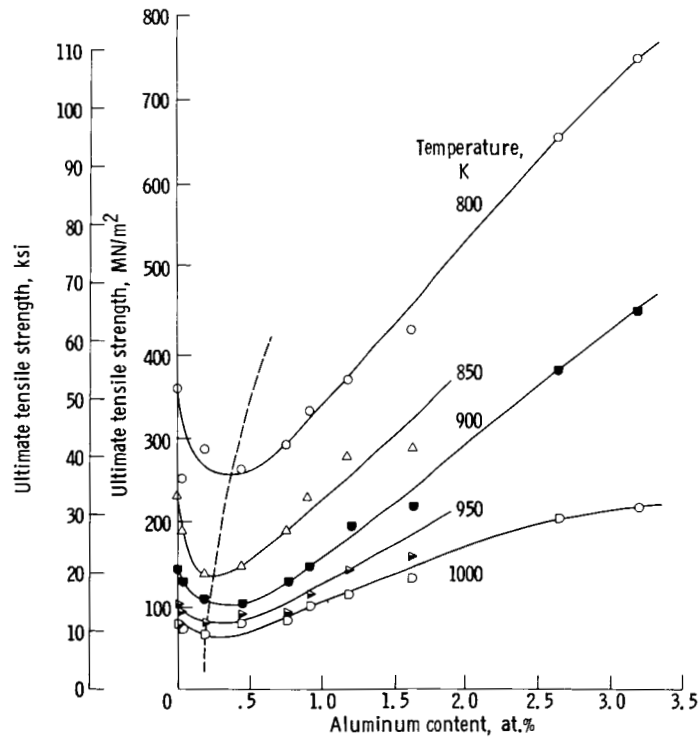


Figure 5. - Effect of aluminum content on ultimate tensile strength of TiNi.

centrations, to about 0.75 atomic percent aluminum, significant solid-solution softening of TiNi occurred, as shown in figure 5 (table III). Above 0.75 atomic percent aluminum, normal strengthening of the alloyed TiNi is observed. Its strength was significantly greater than that of unalloyed TiNi. The ratio of tensile strength to specific gravity of several aluminum-containing alloys compared with TiNi and Ti-6242 at elevated temperatures is illustrated in figure 6 (table III).

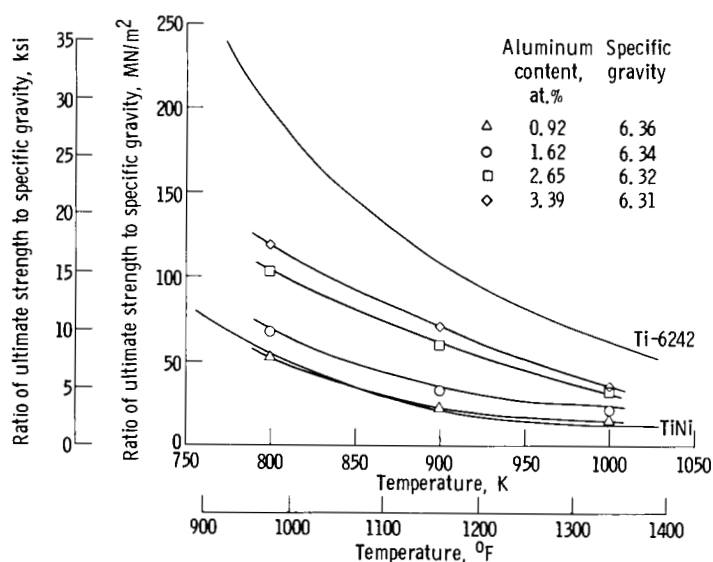


Figure 6. - Effect of aluminum content on specific-gravity-compensated ultimate strength of TiNi.

Figure 7 (table III) illustrates the effect of aluminum content on elongation. Generally, the elongation increased with increasing temperature. However, elongation at the intermediate temperature of 900 K (1160° F) was greater than at any other temperature, almost 58 percent near 1 atomic percent aluminum. Above approximately 2-atomic-percent aluminum, however, the elongation varied with temperature in the expected manner.

The addition of as much as 8-atomic-percent chromium had a moderate strengthening effect on TiNi, figure 8 (table IV). However, the addition of aluminum to TiNi-Cr not only resulted in a further increase in ultimate strength, but also the accompanying minor decrease in density more than doubled the strength-to-specific-gravity ratio of TiNi.

The addition of as much as 4.5-atomic-percent silicon more than doubled the strength-to-specific-gravity ratio of TiNi, as illustrated in figure 9 (table V). Moreover, additions of aluminum to the silicon-containing alloys resulted in the greatest specific-gravity-compensated strength exhibited by any of the alloys investigated, more than triple that of unalloyed TiNi.

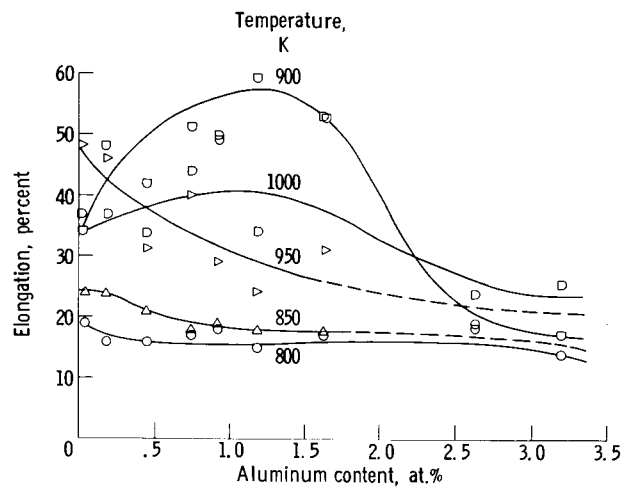


Figure 7. - Effect of aluminum content on elongation of as-swaged TiNi at 800 to 1000 K.

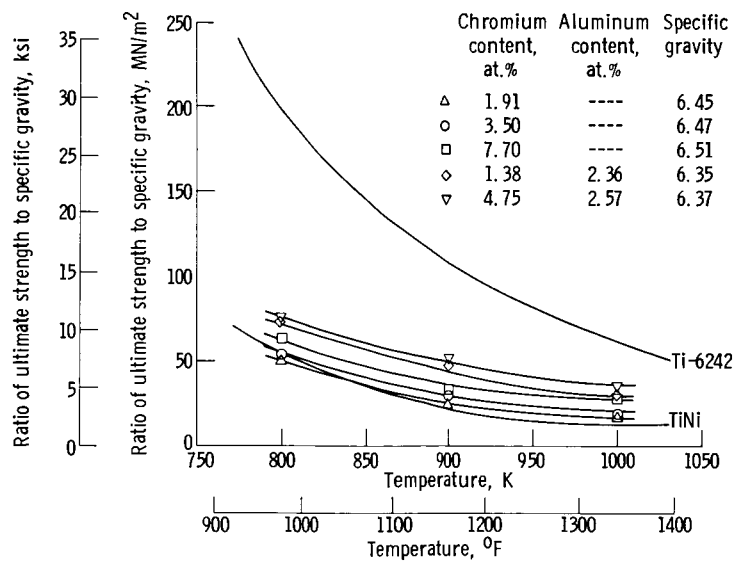


Figure 8. - Effect of chromium and aluminum content on specific-gravity-compensated ultimate strength of TiNi.

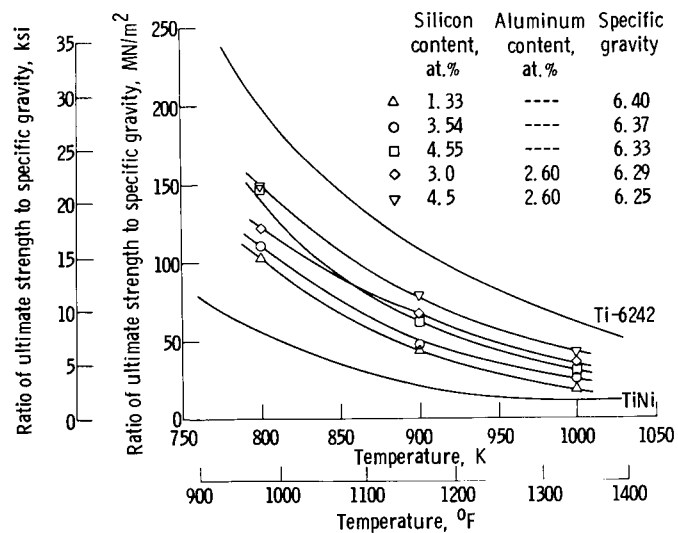


Figure 9. - Effect of silicon and aluminum content on specific-gravity-compensated ultimate strength of TiNi.

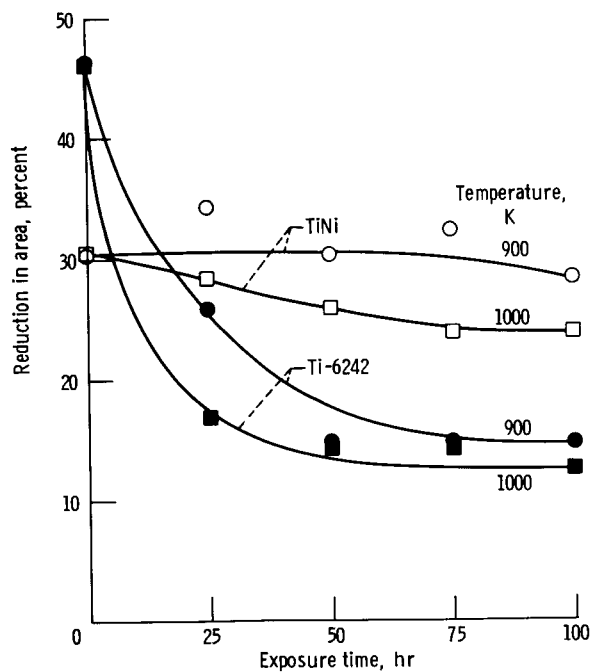


Figure 10. - Effect of elevated-temperature air exposure on room-temperature ductility of TiNi and Ti-6242.

Figure 10 (table VI) illustrates the effect of elevated-temperature air exposure on the room-temperature reduction in area of unalloyed TiNi and Ti-6242. Under the most severe conditions, 100-hour exposure at 1000 K (1340° F), the TiNi retained two-thirds its original ductility, but the Ti-6242 lost almost three-quarters of its ductility.

Figure 11 (table VII) illustrates the drastic effect of alloying on the room-temperature ductility of TiNi. Elevated-temperature air exposure at 1000 K (1340° F) for 100 hours further decreased the ductility, but in general the TiNi alloys were superior in ductility to the Ti-6242.

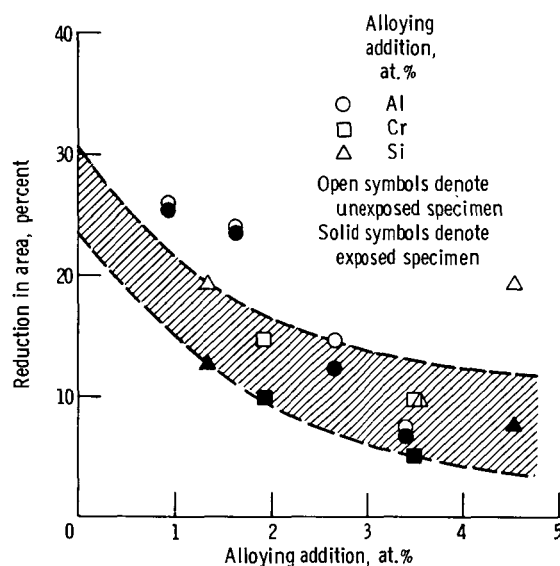


Figure 11. - Effect of alloying on room-temperature ductility of TiNi before and after 1000 K air exposure for 100 hours.

The as-swaged microstructures of the tensile specimens were those of a typical worked structure. This structure persisted after elevated-temperature testing and even after the 100 hours of air exposure at 1000 K (1340° F). Only after a 25-hour exposure at 1250 K (1790° F) under argon was it possible to discern grain boundaries in the microstructure. Figure 12 shows the pearlitic structure that is characteristic of all the TiNi-base alloys, with the amount of pearlite increasing with the amount of alloy addition. The particles that are evident were also present in the worked structure.

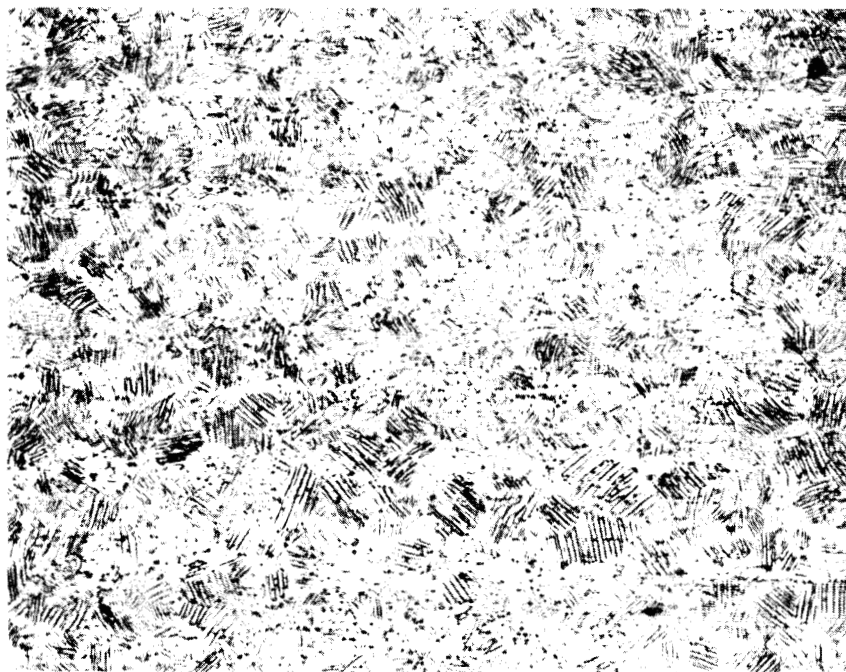


Figure 12. - Microstructure of TiNi - 3.5-atomic-percent-Cr alloy after annealing at 1250 K (1790° F) for 25 hours under argon. Etchant: 30NH₄Cl-50HNO₃-20H₂O. X250.

DISCUSSION

Although little information is available concerning the elevated-temperature mechanical properties of TiNi, studies have been made of its transformation reactions. It is known that the equiatomic compound undergoes a very sluggish allotropic transformation near 900 K (1160° F) (ref. 2) and that above the transformation temperature the structure is simple cubic (CsCl type structure). Controversy exists as to the structure below the transformation temperature. Proposed structures include hexagonal (ref. 3) and simple trigonal (ref. 4), though it is most widely held that the simple cubic structure decomposes into the compounds Ti₂Ni and TiNi₃ (refs. 2, 5, and 6). However, the reaction is so sluggish it will not occur even during furnace cooling, though some metastable precipitate (probably Ti₂Ni₃) does form (ref. 2).

The compound TiNi can be hardened by quenching from approximately 1300 K (1880° F, ref. 7), probably by a martensitic transformation similar to that responsible for the memory-phenomenon near room temperature (ref. 8). Annealing at 900 K (1160° F) causes the formation of Ti₂Ni₃ (ref. 7), probably the nonequilibrium π phase referred to in references 3 and 9. Such transformation reactions would probably not be amenable to elevated-temperature strengthening of TiNi because the decomposition reaction occurs in the temperature range of interest. The transformation does not appear to

affect the strength of TiNi, as illustrated in figure 4.

Alloying had a significant effect on the tensile properties of TiNi. The extent of solid-solution softening exhibited by TiNi with low concentrations of aluminum (fig. 5) decreased with increasing temperature. Nevertheless, softening persisted to almost one-half the melting temperature of TiNi, three times the homologous temperature observed for softening in other alloy systems (ref. 10).

Above 0.75-atomic-percent aluminum, increasing alloy content resulted in increased strength, as with the other alloys investigated. The only other anomalous behavior associated with aluminum additions is the unusually high elongation observed at 900 K (1160° F, fig. 7). As this is also the temperature at which the simple cubic structure reportedly decomposes, the high elongation is probably associated with transformation superplasticity (ref. 11).

In general, the addition of aluminum, chromium, and silicon can increase the elevated-temperature ultimate strength of TiNi between two- and threefold. Considerably greater strengths will be required before the compound could be considered as an elevated-temperature structural material, for example, in compressor blade applications. However, the strengths achieved were presumably by simple solid-solution and second-phase hardening. The most potent strengthening mechanism, precipitation hardening, has yet to be investigated.

All the TiNi alloys have similar microstructures. Traces of the pearlitic structure present in figure 12 were observed in all the specimens held at 1250 K (1790° F) and furnace cooled. Consequently, the alloy additions were probably in solution at the annealing temperature, indicating the alloys may be amenable to precipitation hardening.

Because oxygen contamination is a limiting factor in the use of titanium above 800 K (980° F, ref. 12), it will probably also limit the use of TiNi. Nevertheless, to 1000 K (1340° F), the loss in ductility due to embrittlement was proportionately less than that associated with Ti-6242 (fig. 10). In fact, with aluminum present, the TiNi showed virtually no loss in ductility (fig. 11), even though the exposure temperature was far higher than the temperature range of interest.

Although this study has shown that TiNi is amenable to a significant improvement in elevated-temperature tensile properties by means of alloying, it is not the tensile properties of the TiNi alloys that will determine their usefulness as compressor blade materials. Instead it will be their creep and fatigue behavior, as well as their notch sensitivity and resistance to corrosion cracking, as compared to competing materials, that will be the decisive factors. Unfortunately, such information is lacking even for the simple equi-atomic compound.

CONCLUSIONS

The intermetallic compound TiNi was successfully alloyed with aluminum, chromium, and silicon to increase the elevated-temperature tensile strength as much as threefold while retaining a useful level of ductility. These alloys appear sufficiently promising as a result of this study of their tensile properties and oxygen contamination behavior in the temperature range between 800 and 1000 K (980⁰ and 1340⁰ F) to warrant further development work on strength improvement and on the determination of their mechanical properties pertinent to elevated-temperature structural applications. Compressor blades for advanced air-breathing engines are among such applications.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 7, 1973,
501-01.

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TABLE I. - COMPOSITIONS OF
EXPERIMENTAL ALLOYS

Alloy	Al	Cr	Si	Specific gravity
	Analyzed content of alloying addition, at .%			
TNB-1	0.04	----	----	6.45
TNB-2	.19	----	----	----
TNB-3	.45	----	----	----
TNB-4	.75	----	----	----
TNB-6	.92	----	----	6.36
TNB-7	1.18	----	----	----
TNB-8	1.62	----	----	6.34
TNB-19	2.65	----	----	6.32
TNB-21	3.39	----	----	6.31
TNC-2	----	1.91	----	6.45
TNC-3	----	3.50	----	6.47
TNC-4	----	7.70	----	6.51
TNC-8	2.36	1.38	----	6.35
TNC-9	2.56	4.75	----	6.37
TNE-3	----	----	1.33	6.40
TNE-4	----	----	3.54	6.37
TNE-5	----	----	4.55	6.33
TNE-8	2.60	----	2.85	6.29
TNE-9	2.60	----	4.50	6.25

TABLE II. - TENSILE STRENGTH OF AS-SWAGED TiNi
AND Ti-6242 AT ELEVATED TEMPERATURES

Alloy	Specific gravity	Test temperature		Ultimate strength	
		K	°F	MN/m ²	ksi
TiNi	6.45	800	980	361	52.5
		850	1070	232	33.7
		900	1160	142	20.6
		950	1250	103	14.9
		1000	1340	84	12.3
Ti-6242	4.54	800	980	917	133
		850	1070	672	97.5
		900	1160	504	73.1
		950	1250	399	58.8
		1000	1340	263	38.2

TABLE III. - ELEVATED-TEMPERATURE TENSILE PROPERTIES OF TiNi
ALLOYED WITH ALUMINUM

Alloy	Aluminum content, at .%	Test temperature		Ultimate strength		Elongation, percent
		K	°F	MN/m ²	ksi	
TNB-1	0.04	800	980	253	36.7	19
		850	1070	188	27.3	24
		900	1160	128	18.6	34
		950	1250	94.5	13.7	48
		1000	1340	78.6	11.4	37
TNB-2	0.19	800	980	283	41.0	16
		850	1070	136	19.7	24
		900	1160	105	15.3	48
		950	1250	80.0	11.6	46
		1000	1340	66.2	9.6	37
TNB-3	0.45	800	980	262	38.0	16
		850	1070	146	21.2	21
		900	1160	103	14.9	42
		950	1250	94.5	13.7	31
		1000	1340	90.3	13.1	34
TNB-4	0.75	800	980	291	42.2	17
		850	1070	188	27.3	18
		900	1160	125	18.2	51
		950	1250	91.7	13.3	40
		1000	1340	81.4	11.8	44
TNB-6	0.92	800	980	331	48.0	18
		850	1070	232	33.7	19
		900	1160	145	21.0	49
		950	1250	112	16.3	29
		1000	1340	97.9	14.2	49
TNB-7	1.18	800	980	366	53.1	15
		850	1070	277	40.2	18
		900	1160	192	27.8	59
		950	1250	141	20.4	24
		1000	1340	112	16.3	34
TNB-8	1.62	800	980	427	62.0	17
		850	1070	285	41.4	17
		900	1160	217	31.4	53
		950	1250	157	22.7	31
		1000	1340	131	19.0	53
TNB-19	2.65	800	980	656	95.1	19
		900	1160	379	54.9	18
		1000	1340	200	29.0	24
TNB-21	3.39	800	980	751	109	14
		900	1160	450	65.2	17
		1000	1340	221	32.1	26

TABLE IV. - ELEVATED-TEMPERATURE TENSILE STRENGTH OF TiNi

ALLOYED WITH CHROMIUM AND ALUMINUM

Alloy	Cr	Al	Specific gravity	Test temperature		Ultimate strength	
	Content of alloying addition, at. %			K	°F	MN/m ²	ksi
TNC-2	1.91	----	6.45	800	980	323	46.9
				900	1160	159	23.1
				1000	1340	108	15.7
TNC-3	3.50	----	6.47	800	980	348	50.4
				900	1160	186	27.0
				1000	1340	119	17.3
TNC-4	7.70	----	6.51	800	980	414	60.1
				900	1160	197	28.6
				1000	1340	186	27.0
TNC-8	1.38	2.36	6.35	800	980	482	70.0
				900	1160	318	46.2
				1000	1340	180	26.1
TNC-9	4.75	2.56	6.37	800	980	491	71.2
				900	1160	330	47.9
				1000	1340	235	34.1

TABLE V. - ELEVATED-TEMPERATURE TENSILE STRENGTH OF TiNi
ALLOYED WITH SILICON AND ALUMINUM

Alloy	Si	Al	Specific gravity	Test temperature		Ultimate strength	
	Content of alloying addition, at. %			K	°F	MN/m ²	ksi
TNE-3	1. 33	----	6. 40	800	980	664	96. 3
				900	1160	282	41. 1
				1000	1340	124	18. 0
TNE-4	3. 54	----	6. 37	800	980	696	101
				900	1160	296	42. 9
				1000	1340	166	24. 1
TNE-5	4. 55	----	6. 33	800	980	924	134
				900	1160	387	56. 1
				1000	1340	194	28. 2
TNE-8	2. 85	2. 60	6. 29	800	980	758	110
				900	1160	421	61. 1
				1000	1340	219	31. 8
TNE-9	4. 50	2. 60	6. 25	800	980	924	134
				900	1160	498	72. 2
				1000	1340	268	38. 9

TABLE VI. - EFFECT OF ELEVATED-TEMPERATURE
AIR EXPOSURE ON ROOM-TEMPERATURE DUCTILITY
OF TiNi AND Ti-6242

Material	Exposure temperature		Exposure time, hr	Reduction in area, percent
	K	°F		
TiNi	900	1160	0	30
			25	34
			50	30
			75	32
			100	28
	1000	1340	0	30
			25	28
			50	26
			75	24
			100	24
Ti-6242	900	1160	0	46
			25	26
			50	15
			75	15
			100	15
	1000	1340	0	46
			25	17
			50	15
			75	15
			100	12

TABLE VII. - EFFECT OF ALLOYING ON ROOM-
TEMPERATURE DUCTILITY OF TiNi BEFORE AND
AFTER 1000 K AIR EXPOSURE FOR 100 HOURS

Alloy	Content of alloying addition, at. %	Reduction in area, percent	
		Unexposed	Exposed
Alloying addition, aluminum			
TNB-6	0.92	26	26
TNB-8	1.62	24	24
TNB-19	2.65	15	12
TNB-21	3.39	8	8
Alloying addition, chromium			
TNC-2	1.91	15	10
TNC-3	3.50	10	5
Alloying addition, silicon			
TNE-3	1.33	19	12
TNE-4	3.54	10	--
TNE-5	4.55	19	8