

DEVELOPMENT OF THE RHENIUM CONTAINING SUPERALLOY CMSX-4®

FOR SINGLE CRYSTAL BLADE APPLICATIONS IN ADVANCED TURBINE ENGINES

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Summary

A team approach involving several turbine engine companies utilizing the concepts of simultaneous engineering, has been used to successfully develop CMSX-4 alloy for turbine blade applications. The high level of balanced properties determined by laboratory evaluation, has been confirmed during field testing the Solar Mars T-14000 industrial gas turbine with CMSX-4 single crystal (SX) blades in both the coated and bare condition.

## Introduction

Increased operating temperatures and higher rotational speeds resulting in increased component stresses, are primary goals in the continuing development of the gas turbine to provide improved fuel efficiency and power-to-weight performance. Cost reduction, from improvements in turbine component producibility and process yield, and through gains in airfoil component durability, is an additional objective.

The greatest advances in metal temperature and stress capability for turbine blades in the last 30 years has been the result of the development of single crystal superalloy, casting process and engine application technology pioneered by Pratt and Whitney Aircraft (1 thru 19 inclusive). The compositions of the first generation single crystal superalloys which have attained turbine engine application status are shown in Table I. These alloys are characterized by similar creep-rupture strength (density corrected) equating to 45°F (25°C) to 90°F (50°C) temperature capability improvement in terms of time to 1.0% creep, compared to the extensively used directionally solidified (DS) columnar grain alloys MAR M 200 Hf, MAR M 002, MAR M 247 and CM 247 LC® (13). However, they exhibit differing single crystal castability, residual gamma/gamma prime ( $\gamma/\gamma'$ ) eutectic phase content following solutioning, propensity for recrystallization during solution heat treatment, absence or presence of carbides, impact and mechanical fatigue properties (HCF & LCF), environmental oxidation and hot corrosion properties and density.

Carbon has been included in some single crystal alloy compositions to assist vacuum induction refining and alloy cleanliness. However, the carbide interfaces with the metal matrix are surfaces of high energy and can act as crack initiation sites along with fractured carbides during creep deformation and mechanical fatigue loading (20). Casting micropores in NON-HIP'ed single crystal castings can also be of prime importance in determining the mechanical fatigue life. Fatigue cracking initiates at the micropores and the crack initiation can occur quite early in notched specimens. It is reported that the micropores in a single crystal first generation alloy become coated with  $\gamma'$  during high temperature service exposure and fatigue cracks nucleate in the  $\gamma'$  coating the pore (21).

### First Generation Single Crystal Superalloys

Alloy	Nominal Composition, wt. %											Density kg/dm <sup>3</sup>
	Cr	Co	Mo	W	Ta	V	Cb (Nb)	Al	Ti	Hf	Ni	
PWA 1480 (1)	10	5	-	4	12	-	-	5.0	1.5	-	BAL	8.70
René N-4 (2,3)	9	8	2	6	4	-	.5	3.7	4.2	-	BAL	8.56
SRR 99 (5,6)	8	5	-	10	3	-	-	5.5	2.2	-	BAL	8.56
RR 2000 (5,6)	10	15	3	-	-	1	-	5.5	4.0	-	BAL	7.87
AM1 (8)	7	8	2	5	8	-	1	5.0	1.8	-	BAL	8.59
AM3 (19)	8	6	2	5	4	-	-	6.0	2.0	-	BAL	8.25
CMSX-2 (11,14)	8	5	.6	8	6	-	-	5.6	1.0	-	BAL	8.56
CMSX-3 (11,14)	8	5	.6	8	6	-	-	5.6	1.0	.1	BAL	8.56
CMSX-6 (15)	10	5	3	-	2	-	-	4.8	4.7	.1	BAL	7.98
AF 56 (7)	12	8	2	4	5	-	-	3.4	4.2	-	BAL	8.25

**Table I**

### Second Generation Single Crystal Alloys

	Nominal Composition, wt. %											Density kg/dm <sup>3</sup>
	Cr	Co	Mo	W	Ta	Re	Al	Ti	Hf	Ni		
CMSX-4 (23)	6.5	9	.6	6	6.5	3	5.6	1.0	1	BAL	8.70	
PWA 1484 (17)	5	10	2	6	9	3	5.6	-	1	BAL	8.95	
SC 180 (36)	5	10	2	5	8.5	3	5.2	1.0	.1	BAL	8.84	
MC2 (24)	8	5	2	8	6	-	5.0	1.5	-	BAL	8.63	

**Table II**

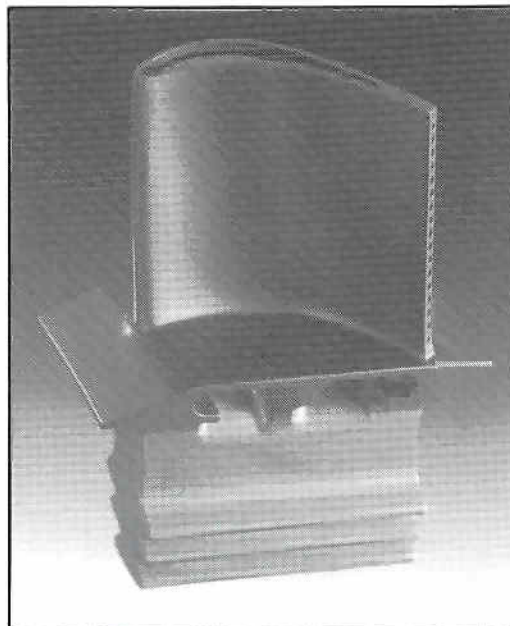
CMSX-2®, CMSX-3®, CMSX-4®, CMSX-6®, CM 247 LC® are registered trademarks of the Cannon-Muskegon Corporation.

MAR M is a trademark of Martin Marietta Corporation.

Turbine engine experience with the first generation single crystal alloys has resulted in process developments being combined with second generation alloy development, to improve and maximize overall properties of the turbine airfoil components (23). Microstructures can be optimized to be fully solutioned and HIP'ed, to contain neither  $\gamma/\gamma'$  eutectic phase, nor regions of incipient melting, carbides, nor microporosity (22). The published compositions of several second generation single crystal alloys are shown in Table II.

Allison has reported completion of initial turbine engine testing of a highly advanced, variable cycle core that offers the potential for doubling powerplant hot-section life and reducing high-pressure turbine weight by 30% (25). Eventually this IHPTET engine is targeted to double engine thrust-to-weight ratio from the best current (10:1) to 20:1, while cutting fuel consumption by 38%. The engine features a turbine wheel with blades fabricated from Lamilloy® single crystal materials; the Lamilloy quasi-transpirational cooling scheme offers a 30% cooling air reduction over advanced film-cooled blades of comparable life (25).

CMSX-4 alloy is a second generation single crystal superalloy containing 3% Re. It has been extensively developed to maximize overall properties, through collaborative programs with several turbine engine companies, involving close to one hundred 400 lb. (182 kg) heats and seven 8000 lb. (3630 kg) production size heats. The alloy's aim chemistry and heat treatment (including the HIP option) have been developed to optimize microstructure and effect low levels of residual microsegregation.



Solar Mars T-14000  
Single Crystal 1st Blade  
CMSX-4 Alloy

Figure 1

### CMSX-4 Superalloy

#### Chemistry

CMSX-4 alloy, developed using a multi-dimensional approach (23) over a ten year period, achieves a high level of balanced properties. The alloy is derived from CMSX-2® alloy (11, 14) and employs the beneficial strengthening effects of Re. The nominal composition and density are shown in Table II.

Lamilloy® is a registered trademark of Allison Gas Turbine Division (GMC).

It is known that Re partitions mainly to the  $\gamma$  matrix, retards coarsening of the  $\gamma'$  strengthening phase and increases  $\gamma/\gamma'$  misfit (26). Atom-probe micro-analyses of Re containing modifications of PWA 1480 and CMSX-2 alloys reveal the occurrence of short range order in the  $\gamma$  matrix (27, 29). Small Re clusters (approximately 1.0 nm in size) are detected in the alloys. The Re clusters act as efficient obstacles against dislocation movement in the  $\gamma$  matrix compared to isolated solute atoms in solid solution and thereby play a significant role in improving alloy strength. Some studies have shown that approximately 20% of the Re in this type of alloy partitions to the  $\gamma'$  (28), thereby strengthening the  $\gamma'$  phase.

The aim chemistry optimization phase for CMSX-4 alloy targeted maximization of creep-rupture response of the alloy utilizing multi-step 99%+ solution heat treatment procedures and ensuring alloy microstructural stability. Extensive experience confirms that fully solutioned microstructures may be readily attained with CMSX-4 alloy airfoils in production vacuum heat treat furnaces with no incipient melting.

### Alloy Melting

The optimized vacuum induction refining (VIR) procedures developed for CMSX-2 and CMSX-3<sup>®</sup> alloys and discussed in (11), were used to produce the seven CM V-3 furnace 8000 lb. (3630 kg) heats of CMSX-4 alloy, melted to date. Table III shows the C, S, [N] and [O] contents of these heats. Studies by several SX casters demonstrate that high [N] and [O] levels in single crystal superalloy ingot adversely affect SX casting yield due to grain defects (30). The presence of [O], [N] and S master alloy impurities are known to transfer non-metallic inclusions, such as aluminum oxide and nitrides and sulfides of tantalum and titanium, to SX parts (31). Techniques have been developed to successfully recycle CMSX-4 alloy foundry revert.

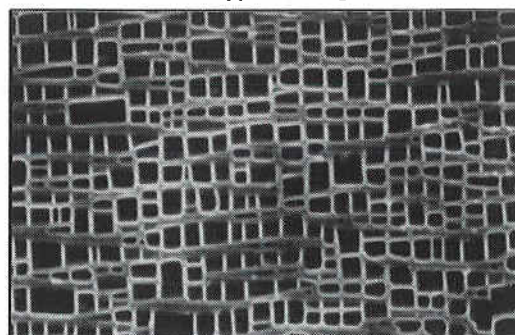
The extensive development of the vacuum induction refining process for the CMSX-4 alloy ingot combined with clean single crystal casting processes have resulted in both clean alloy and airfoil castings in terms of stable oxide dross or refractory inclusions. This is confirmed by production of tens of thousands of single crystal turbine blade castings ranging from .010" (.25 mm) thick Lamilloy crystalfoils to large shrouded turbine blades for the new 80,000 lb. thrust commercial turbofan engines, with attendant high process yields and low levels of grain defects (23).

Heat	CMSX-4 Alloy 8000 lb. (3630 kg) V-3 Furnace 100% Virgin Heats			
	C ppm	S ppm	[N] ppm	[O] ppm
V 7927	20	6	3	1
V 8053	19	5	2	1
V 8054	15	6	3	1
V 8154	21	3	2	2
V 8194	17	4	2	2
V 8195	18	4	2	2
V 8256	15	2	1	1

**Table III**

CMSX-4 Alloy (V 8154) 2nd Stage Solid Blade SX Cast by Allison.

CM 99%+ Soln. AC + 6 hrs/2085°F (1140°C) AC  
+ 20 hrs/1600°F (871°C) AC.  
LE Airfoil Upper Longitudinal



1  $\mu$ m

Laboratory Fully Heat Treated Airfoil  
 $\gamma'$  Microstructure (SEM)

**Figure 2**

## Microstructure

Ageing heat treatment studies have been undertaken which show that the maximum creep-strength throughout the 1500°F (816°C) - 2100°F (1149°C) testing range for (001) orientation specimens, is attained with an average 0.45  $\mu\text{m}$  cubic  $\gamma'$  aligned structure, with a 2085°F (1140°C) high temperature ageing treatment being required. The relatively high ageing temperature suggests CMSX-4 has low  $\gamma/\gamma'$  mis-fit at high temperatures. A typical fully heat treated SEM microstructure for a turbine airfoil is shown in Fig. 2. The solution treatment was undertaken in a laboratory tube furnace finishing at 2 hrs/2410°F (1321°C)/Air Cool (AC). This necessitates a 6 hrs/2085°F (1140°C)/AC age to achieve the desired  $\gamma'$  size. A final age of 20 hrs/1600°F (871°C)/AC is employed. Commercial single crystal turbine airfoils are vacuum solution treated utilizing a Gas Fan Quench (GFQ) from the solutioning temperature, which only necessitates a several hour 2085°F (1140°C)/GFQ high temperature age to achieve the required  $\gamma'$  structure.

TEM foils from a laboratory fully heat treated CMSX-4 airfoil are shown in Figs 3 and 4 (32). Fig 3 in an ordering reflection, shows that the  $\gamma'$  is variable in size locally and by factors (linearly) of about 3. Also, the intervening  $\gamma$  has similar variations in thickness with the formation of secondary, ultra fine  $\gamma'$  in the broader  $\gamma$  gaps resulting from the final 1600°F (871°C) age. EDX composition profiles of the  $\gamma'$  and  $\gamma$  in the TEM foils show the expected trends with the  $\gamma'$  being rich in (Ni with some Co and Cr) and (Al, Ti, Ta and W). The  $\gamma$  is rich in Ni, Cr, Co - disproportionally larger for Cr relative to Co - as well as of course Re and W. Recent research (33) with TEM foils - (EDX-HP Ge detector) and SRR 99 single crystal alloy (Table 1), show some steep element concentration gradients in the  $\gamma'$  phase persist even with an aged 0.5  $\mu\text{m}$  cubic  $\gamma'$  microstructure. The enrichment of Al in the  $\gamma'$  at the  $\gamma/\gamma'$  interfaces is still large and the reduction of Cr at the interfaces is maintained. The average concentrations of elements in the  $\gamma'$  phase have not changed significantly by the two stage ageing heat treatments employed. Interfacial  $\gamma/\gamma'$  chemistry is thought to be important in influencing creep response.

Laboratory Fully Heat Treated  
CMSX-4 Airfoil  
TEM Microstructure

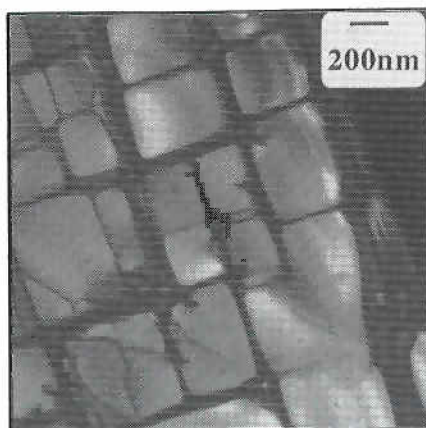


Figure 3

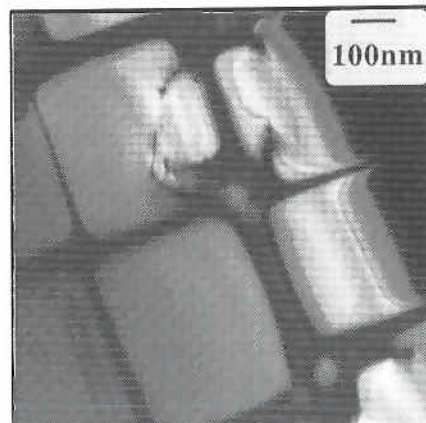


Figure 4

Research (34) has shown that for alloys with a high volume fraction of  $\gamma'$  (at a constant volume fraction), difficult to shear cuboidal  $\gamma'$  precipitates which are spaced as closely as possible will provide optimum creep resistance. Any compositional adjustment which influences the level of misfit between the  $\gamma$  and  $\gamma'$ , the chemistry of the  $\gamma'$ , the equilibrium volume fraction of  $\gamma'$ , or its coarsening behaviour, will have major resultant effect on creep properties, by changing  $\gamma'$  morphology or shape or the matrix gap dimension.

## Creep-Rupture and Phase Stability

The stress-rupture temperature capability advantage of CMSX-4 alloy over CMSX-2/3 alloys is 64°F (35°C), based on density corrected average properties, in the 36 ksi/1800°F (248 MPa/982°C) testing regime (23). The data also suggest that CMSX-4 alloy has useful strength at 2100°F (1149°C) (23). The Larson-Miller stress-rupture data base generated by CM includes over 175 data points from nine heats, including six 8000 lb (3630 kg) heats. These properties were undertaken on both .187" dia. (4.8 mm) test bar and .070" dia. (1.8 mm) machined-from-blade (MFB) specimens, with no fall-off in properties apparent with production heat scale-up to 8000 lbs (3630 kg).

Industrial gas turbine applications for the alloy has necessitated creep-rupture testing out to 4000 hours rupture life at 20 ksi/1800°F (138 MPa/982°C). The 1800°F (982°C) stress-rupture data shown in the log-stress to log-life plot in Fig. 5 shows no fall-off in properties due to undesirable microstructural changes.

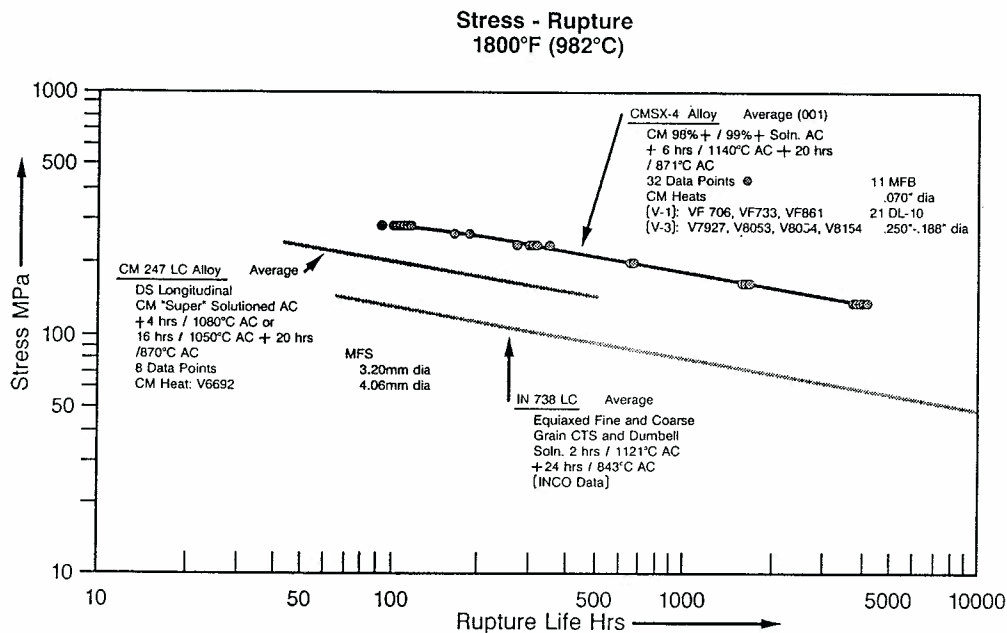


Figure 5

Microstructures of a post-test (.25" dia. (6.4 mm)) specimen creep-rupture tested at 20 ksi/1800°F (138 MPa/982°C)/3921 hrs  $t_r$  are shown in Figs 6 & 7 (32). The generally irregular format of the agglomerated  $\gamma/\gamma'$  structure is apparent. Extremely small (700 nm (0.7  $\mu$ m)) rhombohedral topological intermetallic phases surrounded by  $\gamma'$  can just be discerned at (A) in Fig 7. Analytical TEM shows these phases to be ((Ni, Co) W) (Cr, Re) rich (32). However, no creep cracking has been found to be associated with these phases. It is postulated in (35) that the development of a range of extremely small heterogeneity in Re containing single crystal or DS superalloys during creep can be of fundamental importance to the improvement of creep behavior.

CMSX-4  
 Post-test TEM Microstructure  
 20 ksi/1800°F  
 (138 MPa/982°C)/3921 hrs<sub>t</sub>

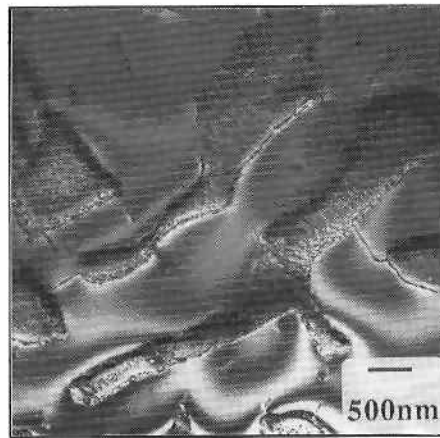


Figure 6

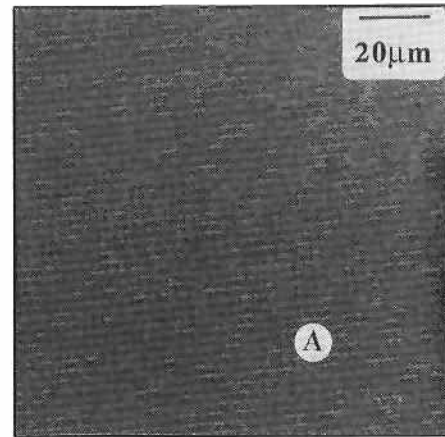


Figure 7

[Optical]

### Mechanical Fatigue

The absence of significant residual  $\gamma/\gamma'$  eutectic phase, carbides, oxide, nitride or sulphide inclusions & microporosity (resulting from the ability to readily fully solution CMSX-4 and HIP) (Figs 8 & 9), results in remarkably high mechanical fatigue properties, with smooth and in particular notched specimens. At 1382°F (750°C) the smooth specimen HCF strength of CMSX-4 to  $10^7$  cycles  $N_f$ , is improved by 50% by HIP. HIP'ing CMSX-4 results in significantly greater mechanical fatigue property improvements compared to that attained with non-Re containing alloys, indicating that the elimination of the micropores is not only the important factor. Increased refractory element (W, Re & Ta) homogeneity may also be relevant. Additionally, the superior creep strength of CMSX-4 alloy over first generation single crystal alloys is expected to be a significant advantage in HCF conditions where sufficient time is spent at stress and temperature to enable creep-fatigue interactions to occur (17).

CMSX-4 Alloy (V 8054) SX Test Bar  
 14mm Diameter. 99%+ Soln. GFQ + IMT HIP  
 15 ksi/2400°F (1316°C)/5 Hrs FC + CM Re-Solnd.  
 3 Hrs/2410°F (1321°C) AC.

Longitudinal

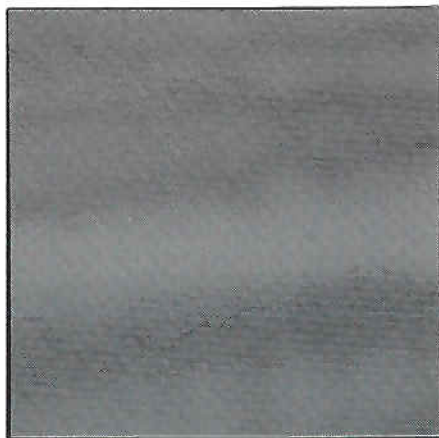


Figure 8

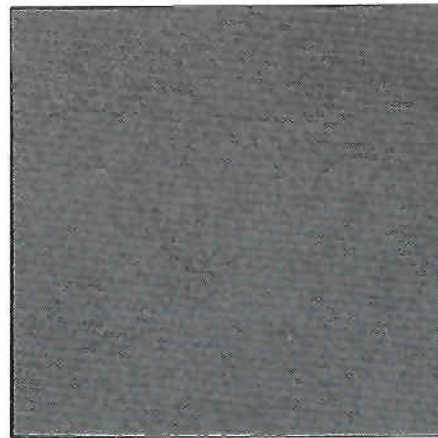
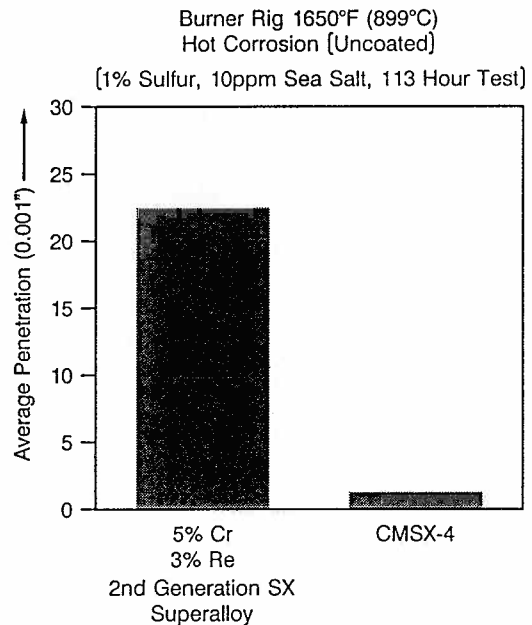


Figure 9

## Oxidation and Hot Corrosion

Cyclic burner rig testing has shown CMSX-4 to have excellent high temperature bare oxidation resistance (23). Burner rig sulphidation testing under a variety of Type I hot corrosion test conditions (e.g. Fig 10) show the alloy to have performance similar to IN 792 in longer term testing.

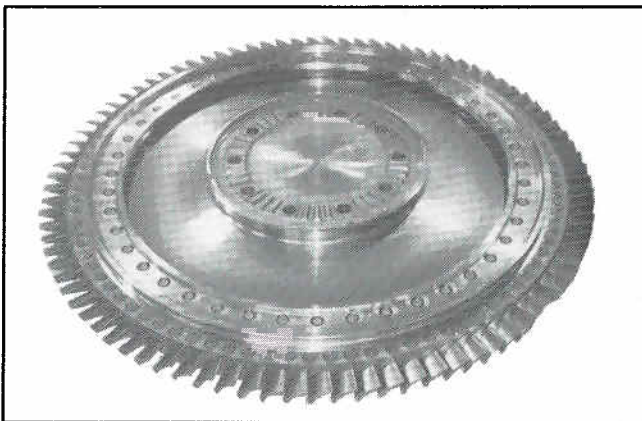


**Figure 10**

## Industrial Gas Turbine Experience

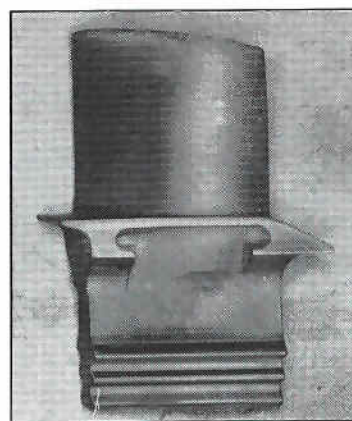
CMSX-4 alloy was introduced for turbine blading in the Solar Turbines Inc. Mars T-14000 engine in 1990. This resulted from the need for increased durability in the uprated Mars engine. The initial engine field test was performed at a natural gas pipeline station where the engine was used to drive a Solar C-601 gas compressor set. In this engine the first stage turbine rotor (Fig 11) had a "rainbow" set of blades with test variables which included blade alloy, (CMSX-4 and equiaxed MAR M 247) coatings, cooling flow and minor blade geometrical differences. The engine was removed for inspection after accumulating 4,333 service hours and 44 starts. The engine was fueled on natural gas.

During tear down inspection it was observed the blades were in good condition. Typical as-received and after-test blades are shown in Fig 1 and Fig 12. Representative components were removed for detailed metallurgical evaluation and replaced with new parts. The remaining hot section components were replaced in the engine with the plan to continue the engine test for a total of 20,000 hours. At the time of writing they have accumulated an additional 4,000-5,000 hours in the engine at full rated conditions.



Solar Mars T-14000 1st Stage  
turbine assembly - CMSX-4 blades

**Figure 11**



Solar Mars T-14000 1st blade in  
CMSX-4 after 4,333 service hours

**Figure 12**



