Effects of Multiple Solution Heat Treatment Cycles on Recrystallization of Single-Crystal Superalloy CMSX-4®

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Understanding the effects of multiple heat treatment cycles on single-crystal superalloy CMSX-4® is imperative to ensure that proper homogenization occurs without introducing grain boundaries that can impair its creep strength in high temperature applications. An experimental matrix was conducted to determine how varying induced plastic strains affect the chance of recrystallization. The team found that even after one solutioning cycle, recrystallization was observed in strains as low as 0.1%. The apparent sensitivity of CMSX-4® to induced strain requires more data to confirm that it is the cause of recrystallization in these samples rather than accidental impact or handling mistakes.

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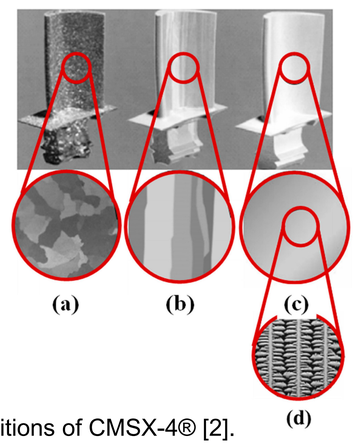
Background

Overview: CMSX-4® is a single crystal nickel-based superalloy that is commonly used in jet turbine blades for its excellent mechanical properties. To obtain its exceptional creep strength, CMSX-4® is cast as a single crystal, removing the inherent weaknesses that can occur along grain boundaries; the alloy is then given a complex heat treatment.

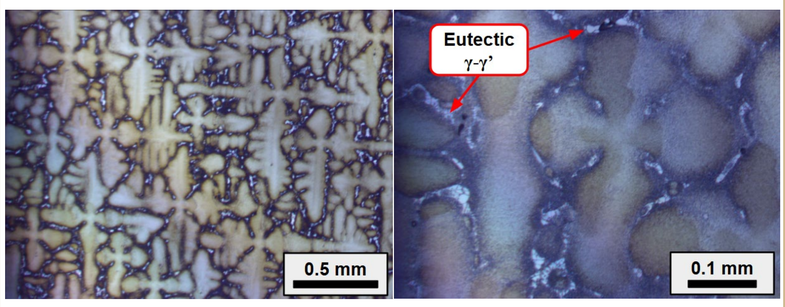
As shown by the image on the right [1], conventionally cast superalloys (a) develop a polycrystalline structure with many grain boundaries. On the other hand, if the blade is directionally solidified (b), grains align along the blade's axis, removing grain boundaries that are perpendicular to the loading of rotating blades. Finally, if grown as a single crystal (c), then grain boundaries are completely removed, though the microstructure remains dendritic (d).



Below is a table of the nominal compositions of CMSX-4® [2].

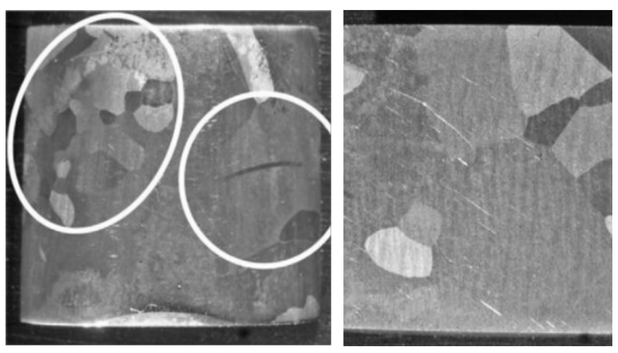
Element	Ni	Al	Ti	Cr	Co	Mo	Ta	W	Re
Wt%	61.8	5.6	1	6.5	9.5	0.6	6	6	3

As-cast Microstructure: In the typical phase change behavior of CMSX-4®, an increase in the level of Al to what is known as γ-Ni will form a second precipitate phase called the γ' phase. During casting, there is not only a precipitation of a γ' phase, but also the formation of a eutectic $\gamma - \gamma$' structure in the interdendritic regions. There is a large focus on these $\gamma - \gamma$' regions because they are detrimental to the creep resistance of CMSX-4®. The microstructural images below were taken from an as-cast etched sample; the eutectic proportion is 5.62% of the sample, as measured by point-counting.



Heat Treatment: To effectively dissolve the γ' constituent back into the dendrites and shrink the eutectic pools, heat treatment is performed in a vacuum to prevent oxidation, and can reach temperatures greater than 1300°C. The heat treatment is performed over 15-20 hours with very slow heating rates to avoid incipient melting. However, sometimes a single solution treatment is not sufficient in decreasing the eutectic constituent below customer specifications, and the part must be solution treated again until the requirements are met. It is in these multiple solution cycles that RX may occur more readily.

Recrystallization (RX): Samples that have experienced plastic strain can recrystallize during a heat treatment cycle, leading to the formation of grains in the microstructure. This plastic strain may occur from constrained contraction during casting, mold removal, impact damage, or handling. Microstructurally, recrystallized grains are typically angular and have a distinct color difference to that of bulk single crystal. The images [3] show examples of RX in CMSX-4® after solution heat treatment. The formation of grains, likely from some strain during casting, means this entire part would have to be scrapped.



Project Objective

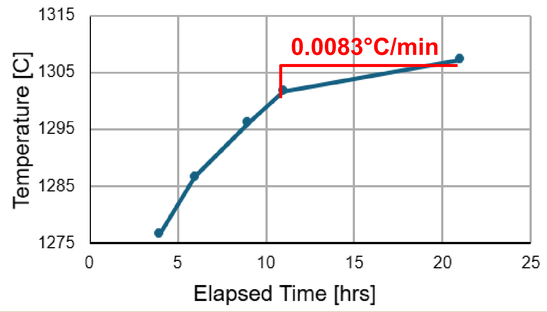
There is limited information available on how re-solutioning cycles impact recrystallization (RX) behavior. The objective of this team was to formulate and execute an experimental design to understand the effect of performing multiple solution cycles on the microstructure of CMSX-4® at minimal strain levels; essentially, to determine the maximum number of cycles that can be allowed in a sample with small amounts of induced strain before recrystallization occurs.

Experimental Procedure

Samples: Howmet provided ½-in diameter cylindrical creep test samples of CMSX-4® in the as-cast condition ([001] orientation), from which thirty ½-in tall samples were cut using wire EDM (WEDM). They were carefully handled as to not induce unintentional strain.

Pre-strain: Samples were plastically strained to varying degrees by compression between hardened steel platens. Displacements were measured directly with a caliper to a significance of 10μm.

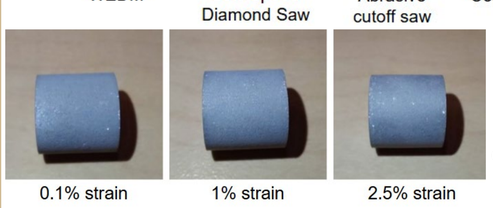
Heat Treatment: Samples were set on an alumina plate in a refractory metal vacuum furnace with an operative pressure of 1×10^{-6} torr. From room temperature, the furnace heated to 1275°C over 4 hours, then slowed substantially, heating only 5°C over the last 10 hours, to avoid incipient melting.



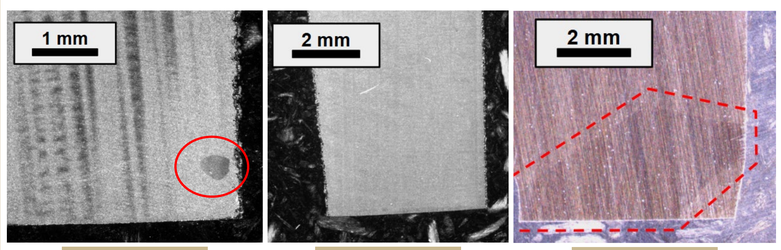
Preliminary Trial

Experimental Design: A preliminary trial on 8 samples was conducted to study how various surface conditions and compressive strains might affect the RX of CMSX-4® after a single heat treatment. The results from the compression samples are specifically meant to inform on the range of compressive strains to use in the main trial.

Condition	Details
WEDM	As supplied by Howmet
Low speed diamond saw	
Abrasive cutoff saw	
Coarse grit	Polished using 320 SiC grinding paper
Fine grit	Polished using 1200 SiC grinding paper
Compression, 0.1%	Compressed in hydraulic press
Compression, 1%	Compressed in hydraulic press
Compression, 2.5%	Compressed in hydraulic press



The 'speckles' on the top and sides of the samples indicate possible RX, though only polishing and etching on a cross-section can reveal grains.



0.1% strain
There was one small grain found on the 0.1% strained sample.

1% strain
No grains were visible in the 1% strained sample, though they might have formed elsewhere on the sample.

2.5% strain
Large grains were observed near the top side of the 2.5% strained sample, evident by their angular shape & discoloration.

Conclusions

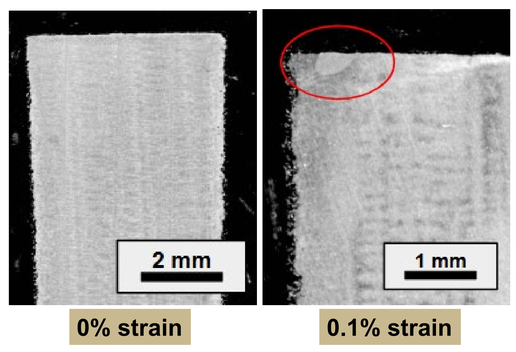
After one heat treatment cycle, the 2.5% compressively strained sample clearly recrystallized, indicating that 0-1% strain was a more suitable range to study RX effects after multiple cycles. Evidence was found of RX at 0.1% strain after a single cycle, further explored in the main trial. Confirmed that coarse cutting or polishing before heat treatment greatly increased the opportunities for RX to occur, especially near the deformed surface.

Main Trial

Experimental Design: A trial consisting of 12 samples was conducted to observe how many heat treatment cycles it took a strained sample to recrystallize. The trial was designed such that a sample from each set would be pulled for characterization after each heat treatment.

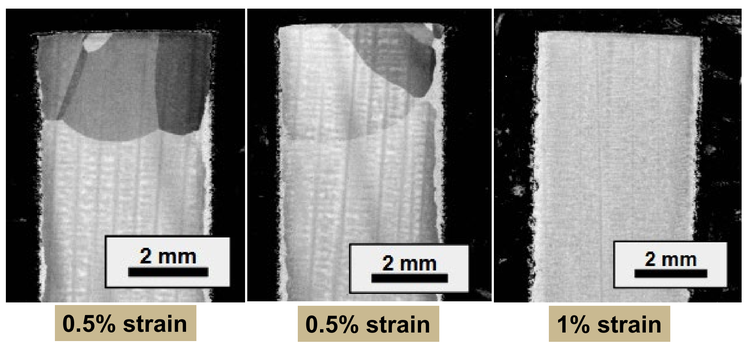
Sample type	Amount
As received, WEDM (0% strain)	3
0.1% Compressive strain	3
0.5% Compressive strain	3
1% Compressive strain	3
Total	**12**

All samples were mounted on their side, so that polishing and etching would reveal RX along the length of the sample.



0% strain **0.1% strain**

After one heat cycle, the 0% strain piece did not show signs of RX, but there is a grain at 0.1% strain and many grains on the edges for the sample at 0.5% strain. Strangely, no grains were visible in the 1% strained sample.



0.5% strain **0.5% strain** **1% strain**

Conclusions

Even from one heat treatment cycle, there is evidence of RX occurring at very small compressive strains, suggesting that similar RX may occur even at small stresses induced during casting. However, the lack of grains in the 1% strained sample suggests either there were alternative stresses on the 0.1% and 0.5% samples from handling or otherwise, or the grains propagated in a different location in the bulk than what was imaged or were removed during polishing.

Discussion and Future Work

From both the Preliminary Trial and the Main Trial, it was confirmed that CMSX-4® may recrystallize after one heat treatment cycle with as low as 0.1% strain, showing that even minimal handling or impact may affect the propagation of grains. The industry practice of casting and heat treating before any parts removal remains the best practice to reduce strain and thus the chances of RX.

An incomplete healing cycle on the second run due to furnace cooling water failure precluded completing the test matrix before the poster production deadline, but the full results should be completed for the final report. With more time and a greater number of samples, further research could be done to quantify the proportion of RX relative to strain and temperature.

Acknowledgements & References

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References

[1] Angel, N. M., & Basak, A. (2020). On the Fabrication of Metallic Single Crystal Turbine Blades with a Commentary on Repair via Additive Manufacturing. *Journal of Manufacturing and Materials Processing*, *4*(4), 101. https://doi.org/10.3390/jmmp4040101
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