

QUENCH CRACKING RESISTANCE OF POWDER METALLURGY SUPERALLOYS

K.-M. Chang

Department of Mechanical and Aerospace Engineering, West Virginia University
P.O. Box 6106, Morgantown, WV 26506 USA

ABSTRACT

Quench cracking behavior of a high strength superalloy, Rene'95, has been investigated by a new approach based on fracture mechanics. A novel laboratory test was set up to identify the criterion for the occurrence of quench cracking. Pre-cracked specimens were loaded by thermal stress induced through the decrease of specimen temperature. When the specimen failed at a certain temperature, on-cooling fracture toughness was measured. Quench cracking toughness, K_Q , was found to be an order of magnitude lower than the room temperature fracture toughness. This brittle fracture at elevated temperature was found to be associated with the intergranular failure of quench cracking. Grain size played a secondary role on quench cracking toughness, and a fine grain structure offered a better resistance to quench cracking than a coarse grain structure.

KEYWORDS

P/M superalloy; quench cracking; intergranular failure; brittle-ductile transition; thermal stress; fracture mechanics; grain size; precipitate solvus.

INTRODUCTION

One of the major challenges in processing large components made of powder metallurgy (P/M) superalloys is to achieve the maximum cooling rate after the solution annealing through various quenching techniques [1-4]. Rapid cooling through the aging temperature range can retain the supersaturation of precipitation hardening elements without forming extensive cooling precipitates. Subsequent aging treatments would then develop a high density of homogeneous precipitates in the alloy matrix and attain a high strength. However, the aggressive cooling from an elevated temperature may result in problems such as severe distortions and excessive residual stresses in components with thick sections. In some cases, unexpected quench cracks may occur during quench process [5].

A fine grain structure of P/M superalloys provides advantages of high strength and low cycle fatigue (LCF) life at low temperatures, but suffers drawbacks of creep strength and fatigue crack propagation (FCP) at elevated temperatures. As the operation temperature of turbine engines increases, a moderate grain size is preferred. New generation P/M superalloys, which received solution anneal at temperatures above the solvus temperature of γ' , were developed in early 1990's [6-8]. Quench process becomes essential to attain the desirable properties in new P/M superalloys. Alloy strength would be lower with annealing above γ'

solvus than that with annealing below γ' solvus. A fast cooling rate from solution anneal is necessary for these P/M superalloys with a moderate grain structure.

In this work, the intrinsic alloy resistance to quench cracking has been investigated in a P/M superalloy, Rene'95, which is known as the strongest superalloy commercially available. Both coarse-grain and fine-grain structures were evaluated by a novel experimental setup that simulated the severest condition of quench process [9]. Quench cracking resistance for P/M superalloys was realized as one of fracture properties that resist high-temperature intergranular failure.

MATERIALS AND EXPERIMENTALS

Rene'95 is one of the earliest P/M superalloys being used commercially [10]. Its tensile strength at 650 °C can reach above 1,500 MPa, which is higher than that of any other superalloy. The nominal composition of P/M Rene'95 is Ni-13Cr-8Co-3.5Mo-3.5W-3.5Al-2.5Ti-3.5Nb-.05Zr-.01B-.06C in weight percent. The alloy can develop about 50% volume fraction of γ' precipitates through appropriate age treatments. The solvus temperature of γ' precipitate is around 1155 °C.

Argon atomization and extrusion compaction of a 20 kg heat were carried out according to the standard procedures for commercial Rene'95 practice [11]. The measured mechanical properties of this laboratory heat met the specification of Rene 95 forging.

Thin sheet single-edge-notched (SEN) specimens were loaded on an Instron hydraulic close-loop machine; the gage section (25.4 mm) was heated by an induction heater. A thermal couple was spot-welded at the center of the specimen to monitor the specimen temperature. The cross section of the gage was 3.175 mm by 1.27 mm. The low thermal mass of this laboratory setup allows rapid heating as well as fast cooling of the gage section. The initial cooling rate from the solution temperature was estimated to be about 100 °C/s. Thermal stress was induced by fixing the displacement of gage length during cooling.

The catastrophic fracture usually occurred within 5 seconds after the quenching started if the quench cracking occurs. Fractography of quench cracks was examined under a scanning electron microscope (SEM) to determine the fracture mode. Metallographic samples were prepared using conventional mechanical grinding and polishing procedures according to the standard laboratory process. An etching solution consisting of 10 ml HCl, 10 ml HNO₃, and 30 ml H₂O₂ was used to reveal grain structure.

RESULTS AND DISCUSSION

Occurrence of Quench Cracking

A metallographic sample of 12 mm × 12 mm × 12 mm cube was cut from P/M Rene'95 forging and solution annealed at 1175 °C. After one hour of heat treatment, the sample was taken from the furnace and water quenched. Many quench cracks formed on the surface, and metallography in Figure 1 shows the intergranular nature of these cracks. The cracks initiated from the edge of the sample and propagated on the surface layer only. In many cases the crack branched as seen in Figure 1. The crack path was always along grain boundaries indicating the intergranular failure.

Examining the fracture surface of a typical SEN specimen showed two distinctive fracture modes: the intergranular fracture for quench crack and the transgranular fatigue failure for pre-crack. A clear beach mark is available for the measurement of pre-crack length, a . The intergranular fracture extends from the beach mark to the other end of the specimen. The observation of intergranular fracture mode confirms that the K_Q value measured by the designed test reflects alloy resistance to quench cracking.

The load curve attained from the thermal stress during cooling shows an abrupt failure without any sign of yielding. This catastrophic nature resembles the shop experience of quench cracking when processing P/M superalloy components. Quench cracks appear in a burst way when the cooling is too aggressive.

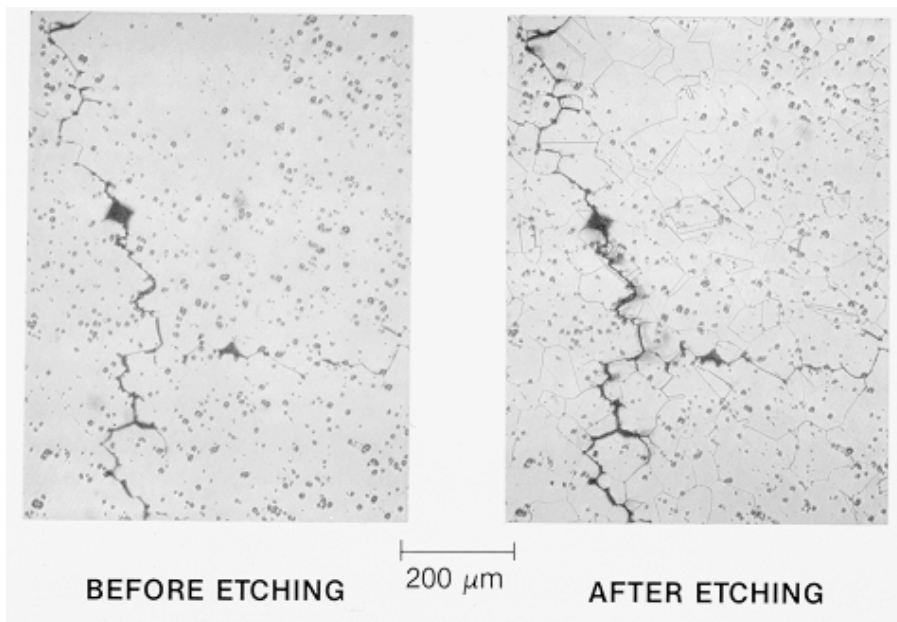


Figure 1
Metallography of quench cracked specimen revealing the nature of intergranular failure.

TABLE 1 lists all tests performed on the SEN specimens. Two solution annealing temperatures were selected in this study: 1175°C for supersolvus annealing, resulted in a coarse grain structure; and 1100°C for subsolvus annealing, resulted in a fine grain structure.

TABLE 1 Quench cracking resistance of powder metallurgy Rene'95 superalloy

Sample ID	Width mm	Thicknes mm	Precrack mm	Start Temp. °C	Fail Temp. °C	Max Load, N	K _Q , MPa√m
Coarse Grain Rene'95 – 1175 °C Annealed							
EX2A2	3.089	1.245	0.775	1180	1040	373.7	7.18
EX2A3	3.134	1.257	0.800	1181	1023	407.0	7.82
EX2A5	3.162	1.308	0.749	1178	1043	420.4	7.19
EX2A6	3.145	1.237	0.749	1123	924	611.2	11.14
EX2A9	3.147	1.267	0.610	1173	1000	560.5	8.32
EX2A10	3.142	1.265	0.267	1171	894	1063.1	9.13
EX2A11	3.165	1.283	1.537	1172	1095	177.9	8.22
EX2A12	3.145	1.295	0.762	1175	900	860.7	15.22
Fine Grain Rene'95 – 1100 °C Annealed							
EX2B1	3.228	1.273	1.461	1124	936	441.3	17.76
EX2B4	3.216	1.275	1.143	1121	N/A	524.9	14.43
EX2B6	3.226	1.273	1.524	1125	N/A	536.0	23.42

N/A: not available because of the failure of thermal couples.

Quench Cracking in Coarse Grains

Solution anneal at 1175°C, a temperature above γ' , dissolves all γ' precipitates in P/M Rene'95. There are MC type carbides randomly dispersed in the matrix, and a coarse grain size of ASTM 7 is observed. Though the forging conditions may affect somewhat the final grain size, P/M superalloys after supersolvus

anneal (above γ' solvus) usually develop grain size of ASTM 6 to 8. The grain size, which is significantly smaller than that of their C&W versions, does not keep increasing with annealing temperature and time.

In total, eight SEN specimens of coarse grain P/M Rene 95 were tested (Table 1). Specimens were heated up to 1175 °C and then cooled under constrain of constant displacement. The maximum thermal stress was reached at the temperature where the catastrophic fracture occurred in each specimen. Figure 2 shows a typical SEM fractography of a broken specimen. A complete intergranular failure occurs during quench cracking; grain boundaries on the fracture surface show very little features except for some carbide particles. No indication of any plastic deformation on the fracture process suggests a brittle nature of cracking.

The failure load was converted to quench cracking toughness, K_Q , for every specimen after the crack length was measured. The measured K_Q values were remarkably low, reflecting the fact of brittle intergranular failure. Typical fracture toughness for P/M Rene 95 is about 70 – 80 $\text{MPa}\sqrt{\text{m}}$ at room temperature.

The dependence of quench cracking fracture on the fracture temperature seems relatively weak, though there is a tendency that the K_Q value increases slightly with the decrease of failure temperature. The thermal stress was primarily determined by the difference of the starting and failure temperatures. To reach a similar K_Q value, the specimen with a short pre-crack length required a high thermal stress. Consequently it would fail at a low temperature, at where the temperature difference is large enough to induce thermal stress required for fracture.

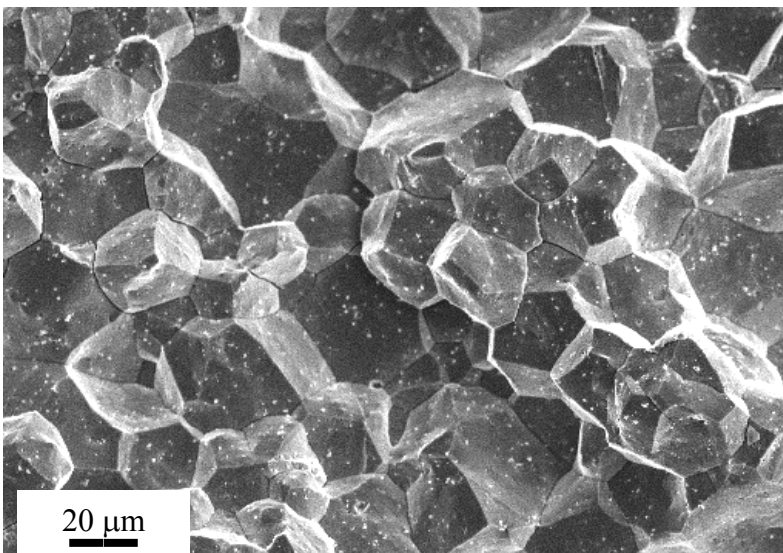


Figure 2
Scanning electron microscopy of intergranular quenching fracture in coarse grain P/M Rene'95.

Quench Cracking in Fine Grains

In the commercial applications, P/M Rene'95 usually receives a solution anneal at some temperature below the γ' solvus. Such a sub-solvus annealing results in a fine grain structure of ASTM 12, which is the typical grain size for most of commercial P/M superalloys. Extensive primary γ' particles with a diameter in micron level are observed. These γ' precipitates formed during the forging process and pin the grain boundaries from coarsening. Such a dual-phase structure exhibits an excellent tensile ductility at high temperatures and is ready for superplastic deformation under the appropriate strain rate [11]. Super-plastic forming through isothermal forging has been employed as a commercial practice to P/M superalloy components. It is of great interest to measure quench cracking toughness of fine grain P/M superalloys.

SEN specimens were prepared from subsolvus annealed P/M Rene 95. After pre-cracking at room temperature, specimens were heated to 1125 °C for quench cracking toughness test. Because of low creep resistance of a fine grain structure at high temperature, there was some difficulty encountered in the setup. Several specimens failed by creep rather than by thermal quench.

In all successful quench cracking tests, specimens failed in the same catastrophic manner as those of coarse grains. The load curve increased linearly until an abrupt drop occurred at some failure temperatures. The results are also listed in Table 1. Two specimens lost their thermocouple during cooling so that no failure temperature was reported. It was expected that the failure occurred at a low temperature for a short pre-crack, and vice versa.

Fracture surfaces of broken specimens were examined under SEM. Intergranular fracture mode occurs through the entire quench crack area as shown in Figure 3. Many micron size primary γ' particles are also observed. This fracture feature is not usually observed since a fine grain structure is always ready to be plastically deformed at elevated temperatures. Grain boundary cleavage indicates a minimum amount of deformation before the failure occurs.

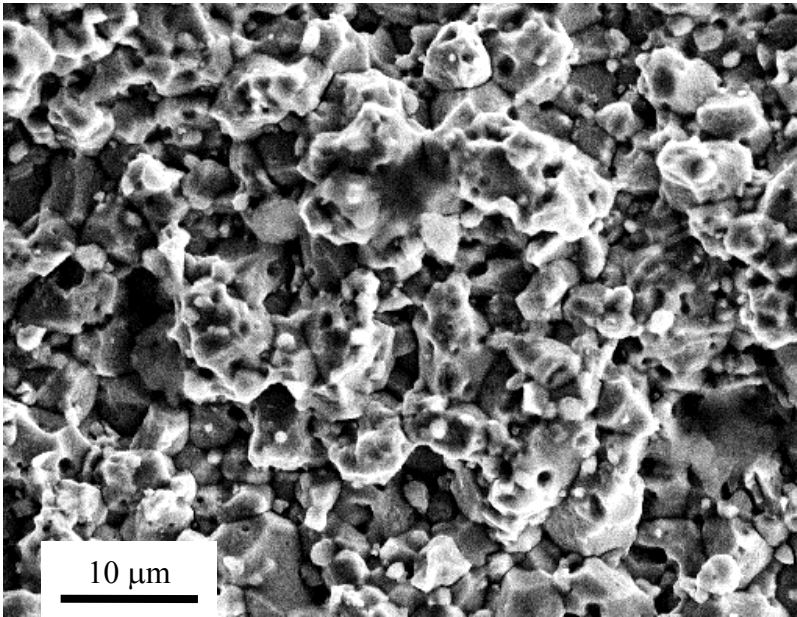


Figure 3
Scanning electron microscopy of intergranular quenching fracture in fine grain P/M Rene'95.

As seen in Table 1, fine grain P/M Rene 95 definitely exhibits a higher K_Q than that of coarse grains. However, these values are far below the room temperature fracture toughness. The brittle nature of intergranular fracture suggested that both fine grains and coarse grains of P/M Rene'95 had a low cohesive energy at elevated temperatures under thermal stress loading. The difference of K_Q values for different grain size is likely attributable to the topographic factor of the crack path. The intergranular cracking would change the direction of crack growth when the crack front encountered the intersection of grain boundaries. A fine grain structure deflected the cracking direction more frequently than the coarse grain structure. Those primary γ' particles on the grain boundaries might also have some beneficial effects.

CONCLUSIONS

A fracture mechanics approach adapted in this study offers a clear understanding on quench cracking behavior of P/M superalloys. Occurrence of quench cracking in the model alloy, P/M Rene'95, is directly related with the resistance to the intergranular fracture at elevated temperatures. Quench cracking, in both fine grain and coarse grain structures, occurs in an intergranular failure.

On-cooling fracture toughness test developed in this work provide a useful method to evaluate the quench cracking resistance. The measured quench cracking toughness, K_Q , in P/M Rene'95 indicated a brittle fracture mode at elevated temperatures, in consistence with the observation of intergranular failure on the fracture surface.

Grain size is not the key factor for the occurrence of brittle fracture at elevated temperatures. In comparison to the coarse grain structure, the final grain structure has a higher quench cracking resistance. However, the K_Q value measured at elevated temperatures is far less than the fracture toughness at low temperature.

ACKNOWLEDGMENTS

The author wishes to thank D.A. Catharine for his technical assistance with experimental works. Helpful discussions with R.D. Kissinger, GE Aircraft Engines, are greatly appreciated.

REFERENCES

1. R.I. Ramakrishnan and T.E. Houson (1992) *JOM*, Vol 44, No 6, p 29-32.
2. J.M. Franchet, F. Devy, P.E. Mosser, Y. Honnort and A. Benallal (1992) In: *Superalloys 1992*, p 73-82, S.D. Antolovich, et. al. (Eds), TMS-AIME.
3. D.R. Garwood, J.D. Lucas, R.A. Wallis and J.Ward (1992) *J. Mater. Eng. Perf.*, Vol 6, p 781-788.
4. R.A. Wallis and P.R. Bhowal (1988) In: *Superalloys 1988*, p 525-534, D.N. Duhal et. al. (Eds), TMS-AIME.
5. R.A. Wallis, N.M. Bhathena, P.R. Bhowal and E.L. Raymond (1988) *Industrial Heating*, Vol 30, January.
6. D.D. Krueger, R.D. Kissinger and R.G. Menzies (1992) In: *Superalloys 1992*, p 277-286, S.D. Antolovich et.al. (Eds), TMS-AIME.
7. K.-M. Chang, M.F. Henry, and M.G. Benz (1990) *JOM*, Vol 42, No 12, 1990, p 29-35.
8. K.-M. Chang (1989) *US Patent* No. 4,816,084,.
9. K.-M. Chang and Boqun Wu (1997) In: *1st International Conf. on Non-Ferrous Processing and Technology*, p 477-481, T. Bains and D. S. MacKenzie (Eds), ASM International.
10. D.R. Chang, D.D. Krueger and R.A. Sprague (1984) In: *Superalloys 1984*, p 245-273, M. Gell et. al. (Eds), TMS-AIME.
11. T.E. Howson, W.H. Couets, Jr. and J.E. Coyne (1984) In: *Superalloys 1984*, p 275-284, M. Gell et. al. (Eds), TMS-AIME.