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Investigation of the Effects of Heat Treating on a Precious Metal Alloy

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Precious Metal Alloy – Paliney 7

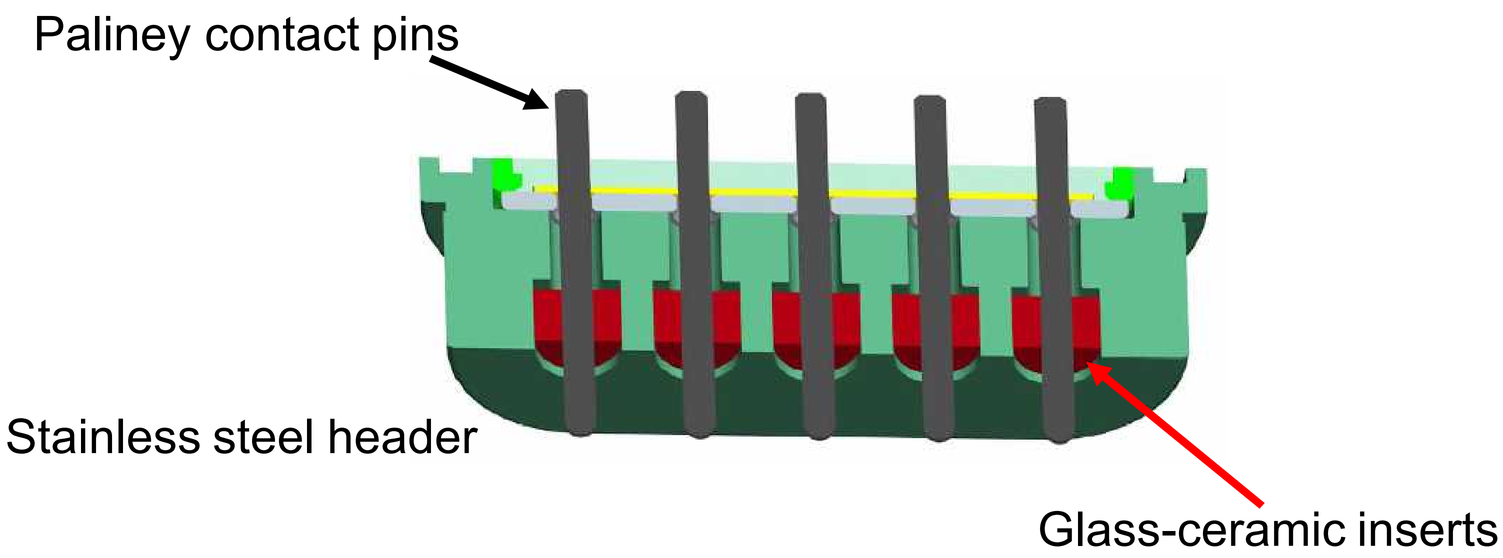
Element	Composition, weight %
Palladium	34.0–36.0
Silver	29.0–31.0
Copper	13.5–14.5
Gold	9.5–10.5
Platinum	9.5–10.5
Zinc	0.8–1.2
Total platinum group metal impurities (iridium, osmium, rhodium, ruthenium)	0.1 max
Total base metal impurities	0.2 max

Composition Range Given for "Pd-based alloy (ASTM B540)"

- High hardness achieved primarily with precipitation hardening
- Corrosion resistant – no plating needed

Application

- Glass-ceramic/metal seal used to hermetically seal several pins into a stainless steel header
- G-C requires special heat treatment
- Heat treatment is not ideal for pin hardness

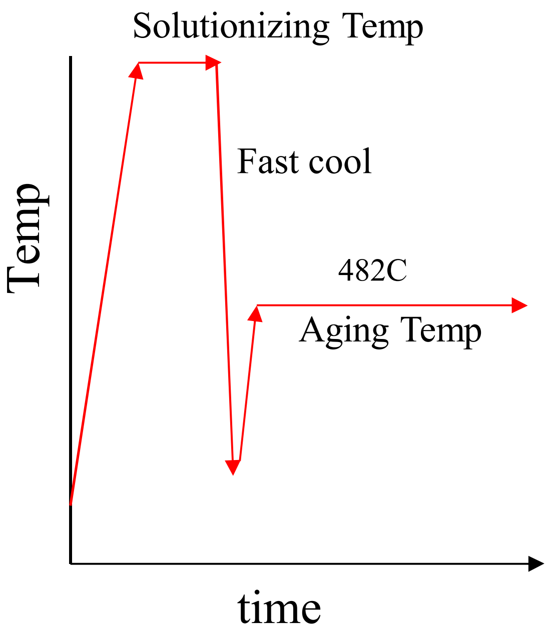


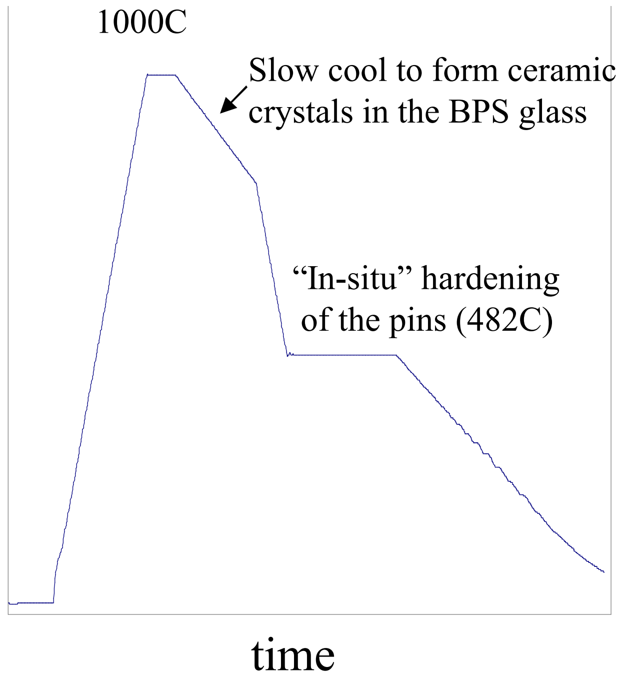
Ideal Age-hardening Process for Paliney vs. "In-situ" age hardening during G-C/metal sealing cycle

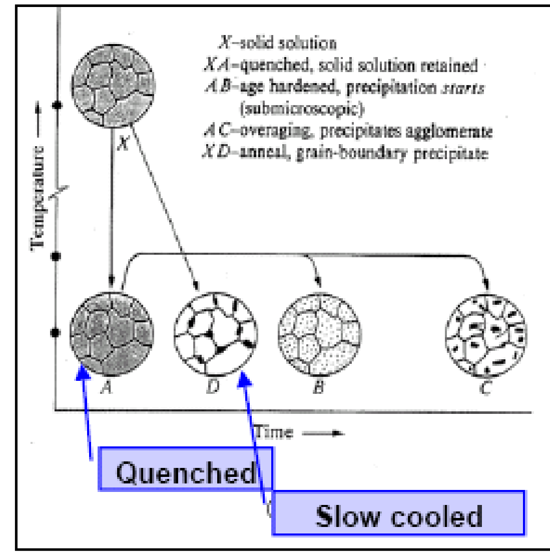
Ideal precipitation-hardening process

A "typical" glass-ceramic seal cycle

Slow cooling can cause non-ideal precipitate microstructure







The slow-cooling rate and long time spent at high temperatures results in a coarse lamellar structure, which causes lower hardness

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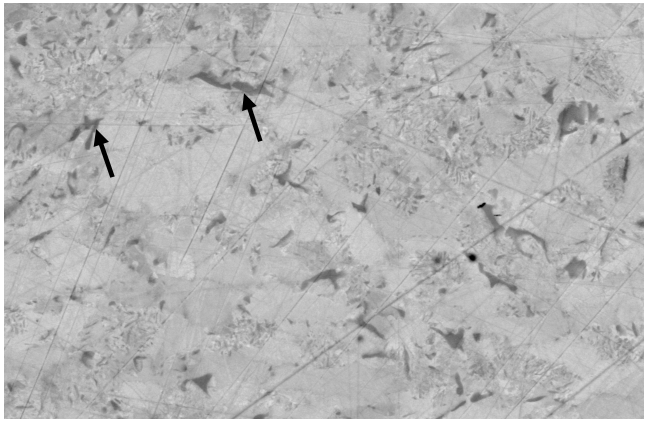
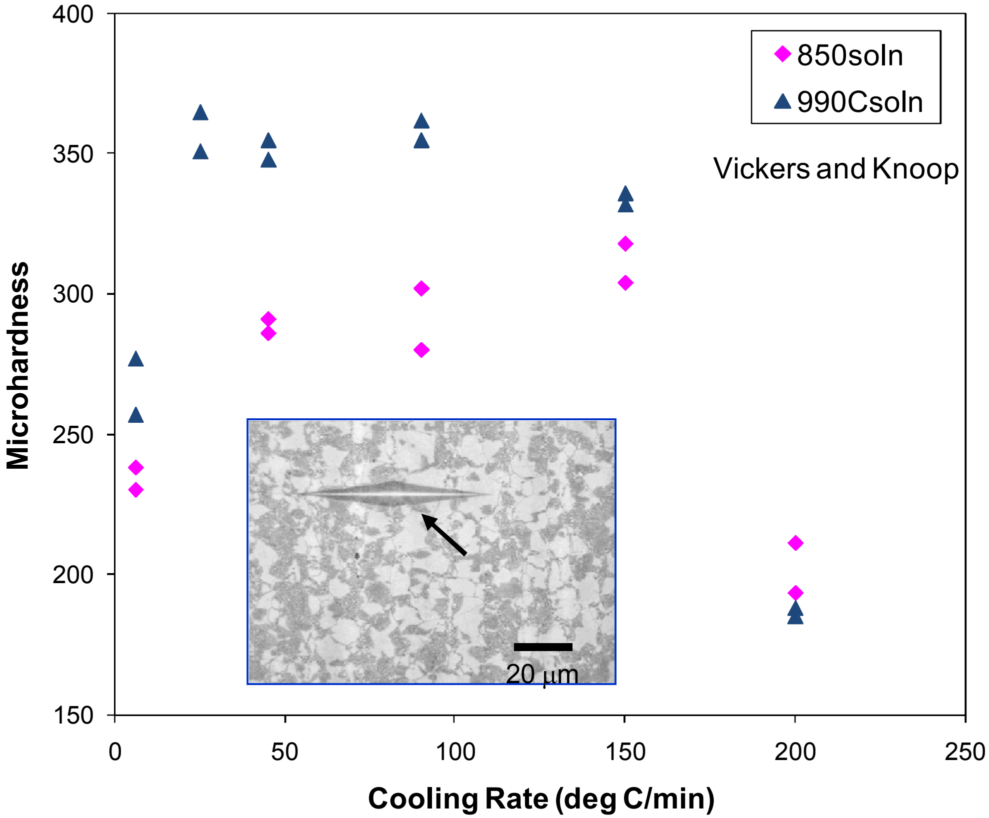
Effect of Cooling Rate on Microstructure and Hardness

- Solutionize pins
  - 850°C to help define low end of solution temp, 990°C to simulate G-C/metal sealing cycle, for 15 min
- Cool at different rates to room temperature
  - 6°C/min to +200°C/min (2 samples used for each rate)
- One pin from each cooling rate subsequently age-hardened
  - Aged at 482°C for 45min
- Microhardness performed on all pins
- Optical microscopy utilized to obtain images of microstructure
- Image analysis performed to quantify amount of precipitate

Hardness Testing – Solutionized Pins



850C "solutionize":
large 2nd phases indicate 850C is not high enough for solution treat



- Vickers and Knoop hardness testing results shown for solutionized pins (not aged)

- Slow cooling rate: coarse, soft precipitates formed, low hardness
- Fast cooling rate: lock in solutionized microstructure, low hardness
- Intermediate cooling rates: not fully solutionized at 850˚C, lower hardness
  solutionized at 990 ˚C, some favorable precipitation upon cooling, higher hardness
- Suggests solvus temperature between 850 ˚C and 990˚C

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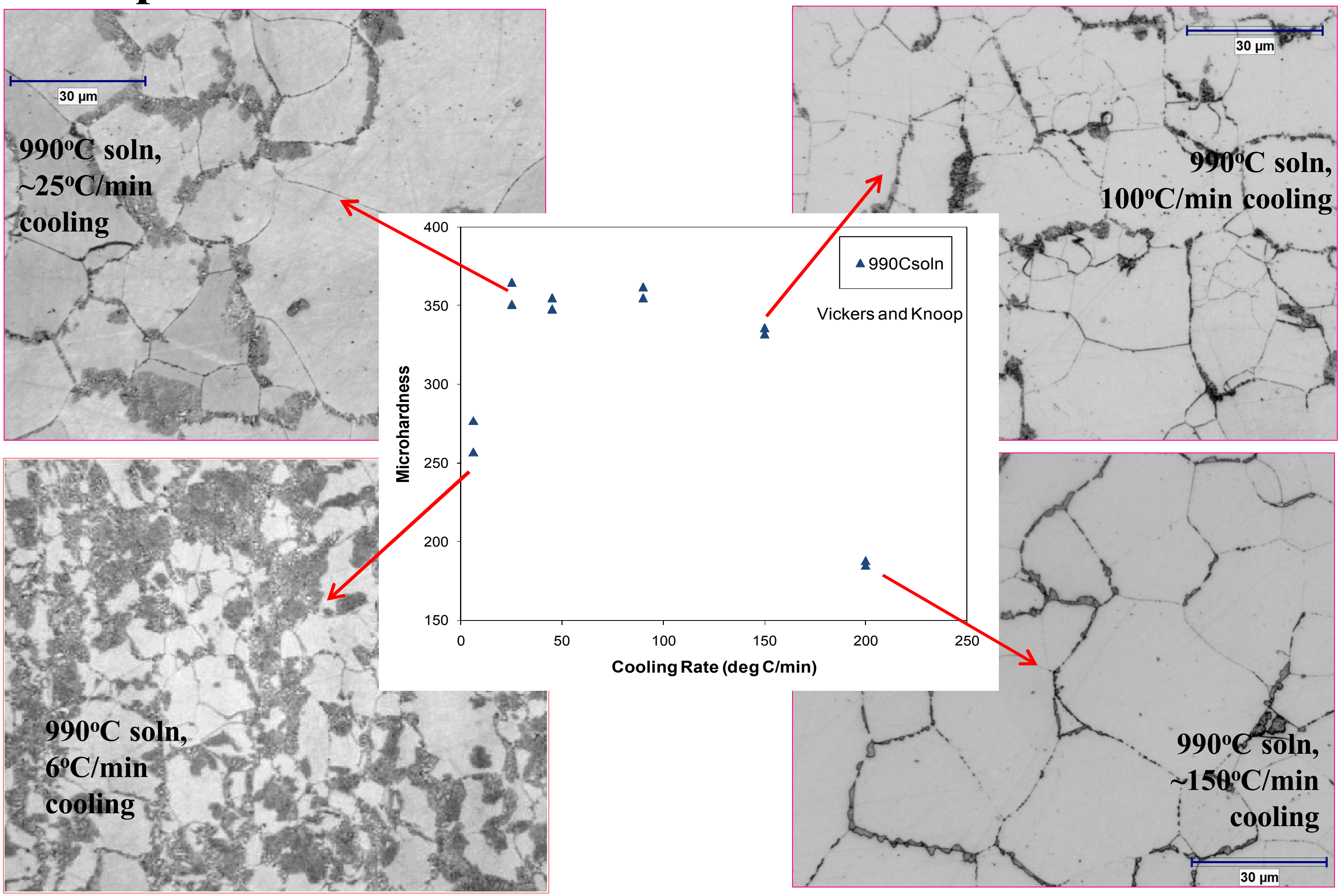
Examples of Microstructure



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Discontinuous Precipitation (DP)

- Precipitation of a 2-phase lamellar structure behind a moving grain boundary
- Recall a eutectoid reaction, in which a single phase transforms into two other solid phases: $\gamma \rightarrow \alpha + \beta$
- Discontinuous precipitation is similar, however this time a single phase transforms into the same phase but with a different composition and an additional phase: $\gamma \rightarrow \gamma' + \beta$

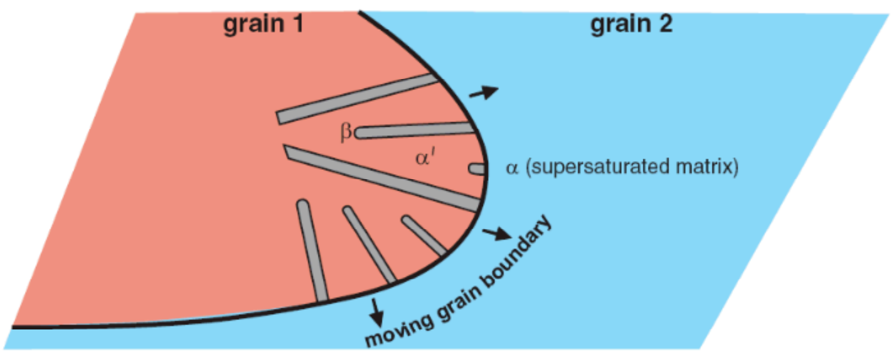

Fig. 1 Schematic of discontinuous precipitation, $\alpha \rightarrow \alpha' + \beta$

Coarse precipitate structure is generally detrimental to mechanical properties as γ' is generally softer than γ

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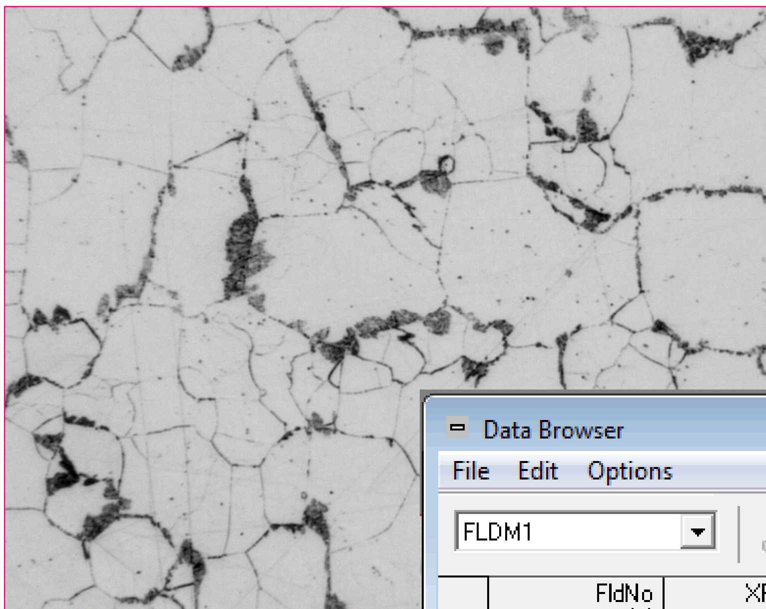
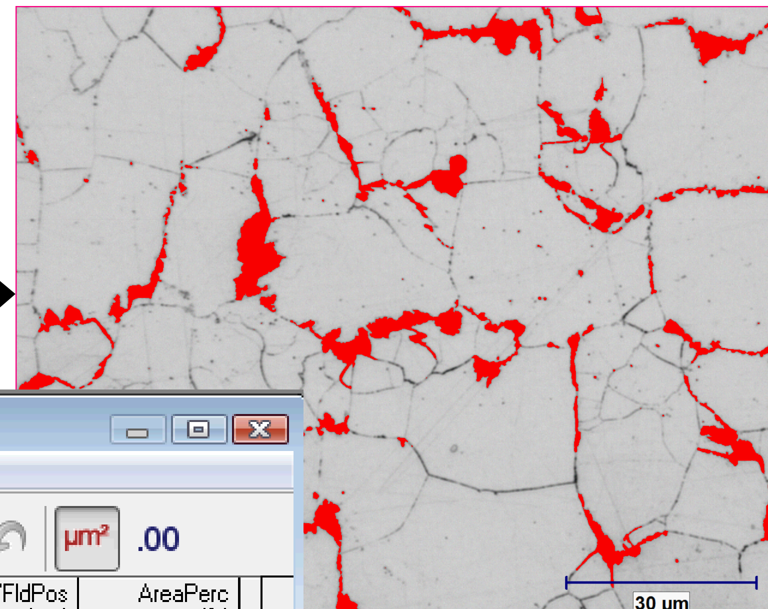
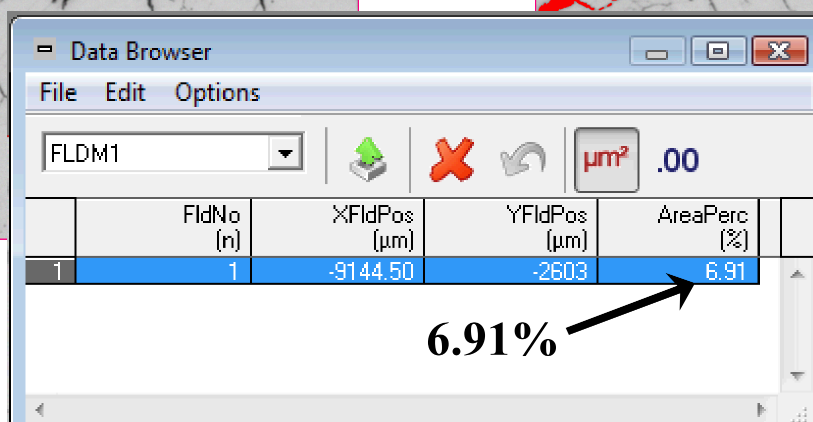
Quantifying Discontinuous Precipitation with Image Analysis

- Clemex Professional Image Analysis Software used to determine area percent of precipitate
- Multiple areas analyzed to give an estimate of volume percent of precipitate for each cooling rate

990^{o}C soln, 100^{o}C/min cooling







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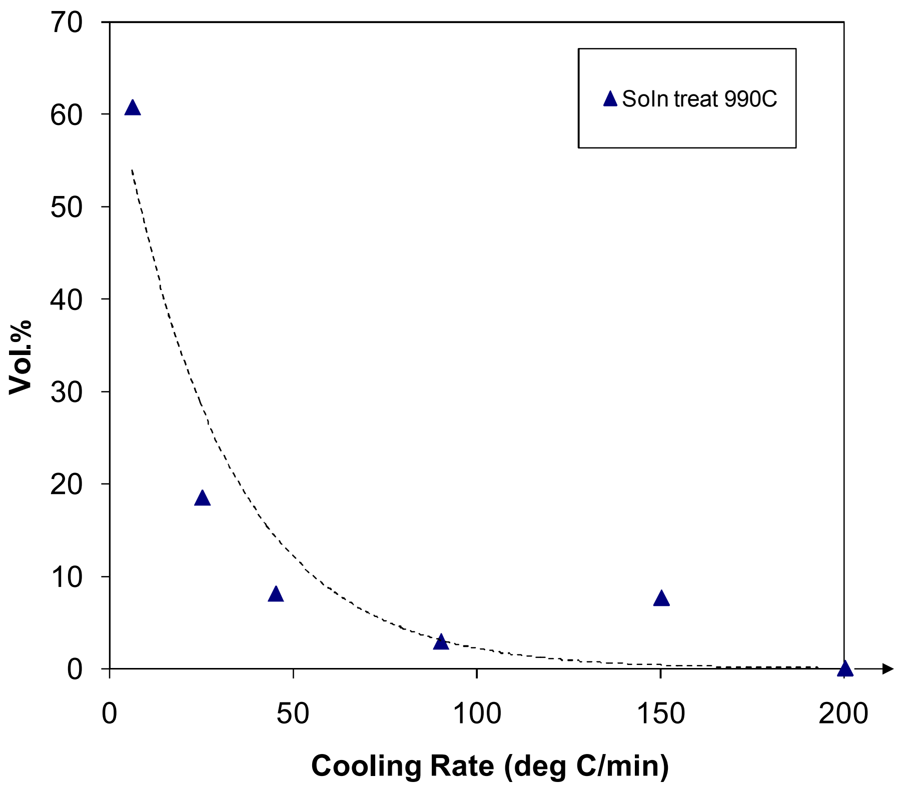
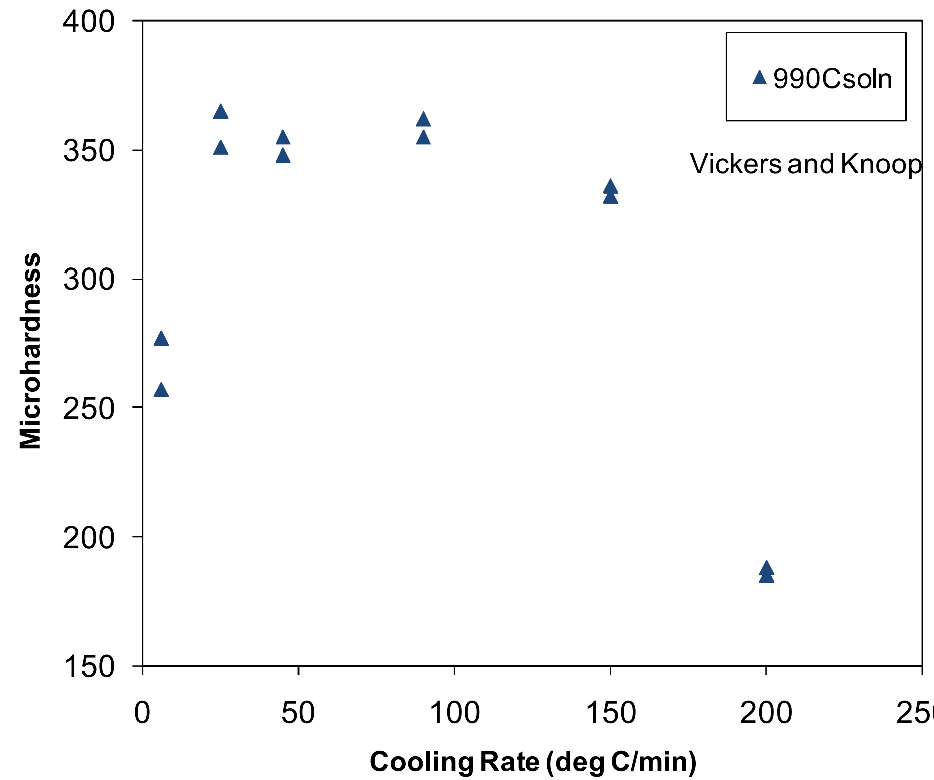
Volume Percent of Precipitate in Microstructure vs. Microhardness





- Very slow cooling rates: high vol % precipitation
- Intermediate cooling rates: some precipitation formation and also age-hardening (fine ppts. not visible with optical microscopy). Low % of DP, not much hardness increase with subsequent aging. In addition, a finer lamellar spacing in the DP regions is expected with faster cooling, thus increasing the hardness of those regions as well.
- Fast cooling rates: no precipitation on cooling. Normal age-hardening precipitation during aging process

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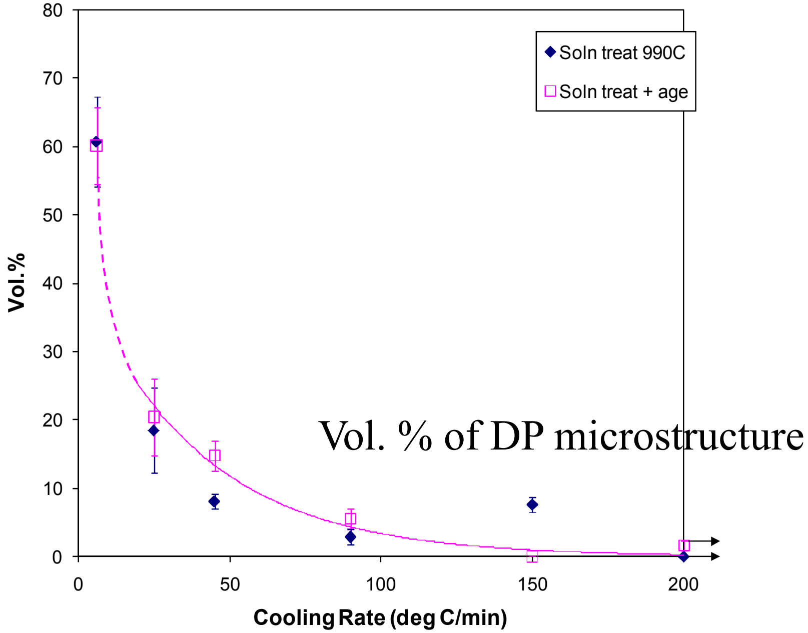
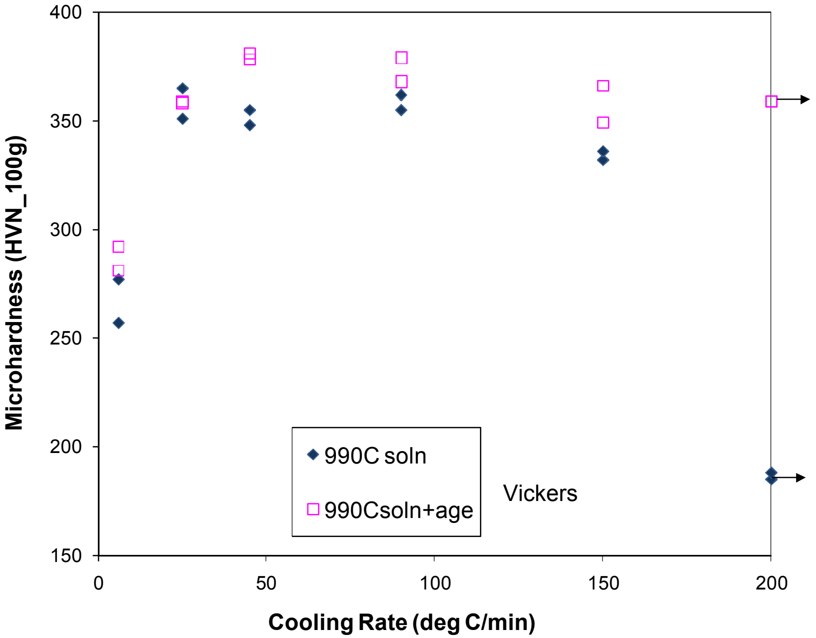
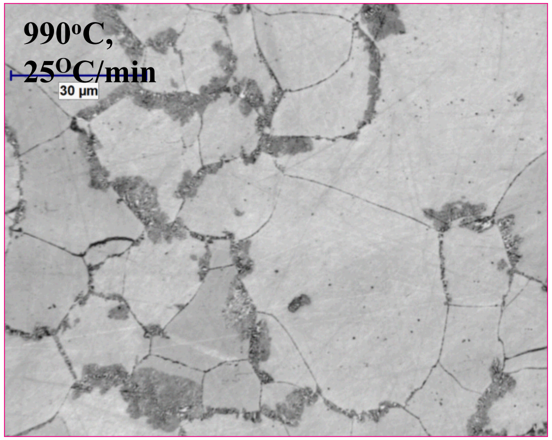
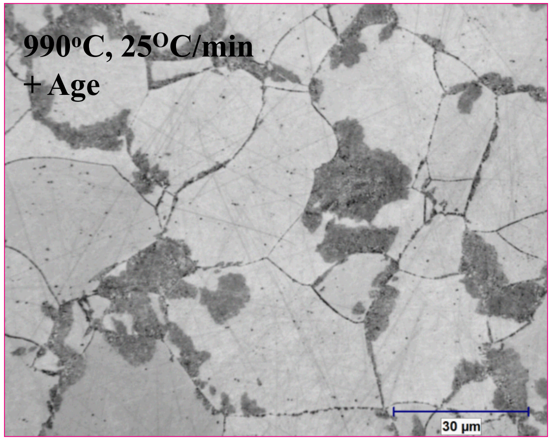
Effect of Aging on Precipitate in Microstructure vs. Microhardness









Aging does not significantly improve hardness in cooling rate range of interest – thus hardness is dominated by vol % of DP

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Conclusions

- Volume % of DP was quantitatively determined using image analysis and correlated to hardness
- Volume % of DP decreases at faster cooling rates
- At slow or intermediate cooling rates, discontinuous precipitation (DP) dominates the hardness achievable in Paliney 7
- These results provide a basis for optimization of glass/metal sealing cycle

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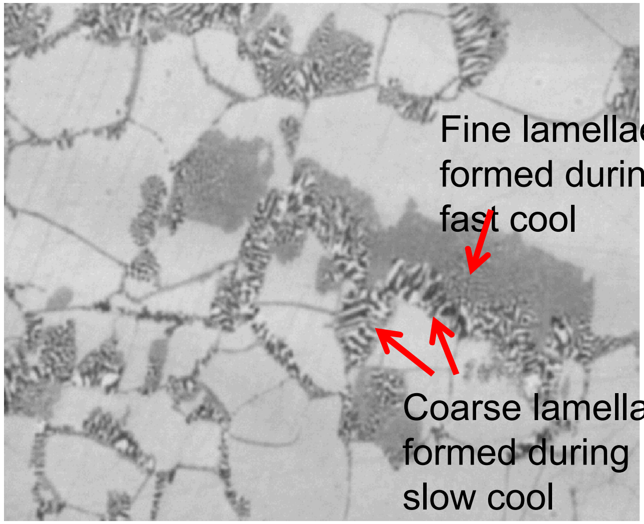
Future Work

- Try to better understand Paliney 7 kinetics to better adjust processing
- Additional microstructural control may be obtained through changes in lamellar spacing with multiple cooling rates
- Creation of a Time-Temperature-Transformation (TTT) diagram using isothermal hold experiments
  - Solution treat at 975°C for 15 min
  - Isothermal hold temps: 800°C, 700°C, 600°C
  - Isothermal hold times: 100s, 1000s, 10000s, 50000s
- Conversion from an TTT to Continuous-Cooling-Transformation (CCT) diagram using methods from Bhadeshia



6C/min cool to 750C, followed by 25C/min cool to RT

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