Hot Deformation Data for Haynes 214, Haynes 230 and Inconel 740H

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This article presents the datasets gathered for the hot processing of three Ni-based superalloys intended for A-USC application, Haynes 214, Haynes 230 and Inconel 740H. Isothermal compression tests were conducted with a Gleeble 3500 at temperatures between 1000 °C and 1200 °C and strain rates between 0.01/s and 1/s to a full true strain of 0.7. The obtained true stress-true strain curves were used as basis for hot processing maps, linking temperature, stress and strain rate. Subsequently, all samples were sectioned through the geometric centre to provide microstructural information, captured using EBSD, as well as EDX for the evolution of secondary phases. Thermodynamic modelling was performed to validate compositions and mass fraction data from EDX measurements. These combined datasets help in understanding the deformation behaviour of a selected range of superalloys, under commercial processing conditions, aiding in process design optimizations and improvements. For complete interpretation of the data the reader should refer to the related publication “Comparative study of the hot processing behavior in advanced Ni-based superalloys for use in A-USC applications”.

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The true stress-true strain curves for are shown in combined graphs for each alloy and strain rate, with all 5 test temperatures in one graph. The EBSD data is split up in bandcontrast and inverse pole figure images, both saved in slides in powerpoint files, one image per processing condition. The property phase diagrams obtained with the ThermoCalc software are split up in separate images, for all stable phases, for the weight fraction and mole fraction of elements within each stable phase, for each alloy based on their as-received composition. Further, the compositions of each stable phase over temperature is saved as excel data.

### Experimental design, materials, and methods

The chamber atmosphere was controlled by evacuating to $1 \times 10^{-2}$ mTorr and subsequent backfilling with research-grade argon gas. The heating rate was 2.5 °C s$^{-1}$, samples were soaked at the test temperature for 30 s, to ensure thermal equilibrium, during which the temperature was maintained. Adequate lubrication between the tungsten-carbide cobalt anvils and the samples was maintained using 0.125 mm thick graphite foil. After the deformation was done, samples remained in the chamber and cooled naturally by heat conduction through the anvils and heat convection through the gas. Using recorded data from attached thermocouples, the cooling rate was determined to be around 60 °C for the first few hundred degrees.
Adiabatic heating corrections have been carried out in order to correct for the effect of additional heat on the flow stress curve, as compared to ideal isothermal conditions. This is an expected effect at higher strain rates, seen by various authors [1–3]. Various correction methods are proposed in the literature, including studies involving numerical solutions using FEM-simulations of hot-compression tests [4] and complex iterative approaches [5]. However, the more commonly used approach is shown in Eq. (5) [6,7].

\[ \Delta T = \eta \alpha \left( \frac{\sigma d\epsilon}{\rho C_p} \right) \]

Here, \( \sigma \) and \( \epsilon \) are the stress and strain values recorded from the compression tests. \( \alpha \) corresponds to the amount of deformation energy transformed into heat, which is usually assumed to be 0.95–0.98, while \( \eta \) stands for the adiabatic correction factor. \( \eta \) is assumed to change between \( \eta = 0 \) for strain rates \( \leq 10^{-3} \text{s}^{-1} \) and \( \eta = 1 \) for strain rates \( \geq 10^1 \text{s}^{-1} \). The integral \( \int \sigma d\epsilon \) represents the area underneath the uncorrected stress-strain curve, \( \rho \) is the material density of 8.05 g cm\(^{-3}\) for Inconel 740H and Haynes 214, and 8.97 g cm\(^{-3}\) for Haynes 230. \( C_p \) is the temperature-dependent heat capacity in J (kg °C\(^{-1}\)), which is defined using a linear fit on the reported specific heat values for the corresponding temperature range from the manufacturer’s datasheet [8–10].

Eq. (5) is generally correcting for the amount of heat transferred to the dies during adiabatic heating. In this dataset the values for \( \eta \) have been defined for each strain rate, based upon comparable values from the literature, i.e. for strain rate of 0.01 s\(^{-1}\) a value of 0.1, for 0.1 s\(^{-1}\) a value of 0.5, and for 1.0 s\(^{-1}\) a value of 0.9 [56, 63, 64]. The flow stress can then be corrected by Eq. (6) [11,12].

\[ \Delta \sigma = \Delta T \left( \frac{\partial \sigma}{\partial T} \right) \]

where by \( \partial \sigma / \partial T \) is obtained by plotting the uncorrected flow stress values at low true strain values (here: \( \epsilon = 0.02 \)) against the temperature, and calculating the first derivative of a linear regression to the curve.

All tested samples were cut in half, parallel to the deformation axis, prior to hot-mounting in conductive resin. Sample preparation followed grinding with silicon carbide and stepwise polishing to a final finish of 0.04 μm with colloidal silica, and subsequent ultrasonic cleaning with ethanol. Analyses were performed on a Zeiss Sigma 30kV FEG-SEM with an Oxford Instruments Nanolys EBSD detector and an SDD EDX detector for chemical analysis. EDX mapping acquisition settings were a 10 keV spectral range, at 2048 channels, and 60–80 m counts per frame, for 2 frames per samples. Elemental EDX maps were processed in ImageJ2/Fiji to optimize contrast and edge sharpness of the precipitates for best particle size detection. EBSD acquisition settings were 8 × 8 and 8 × 16 binning at 15 kV, and maps were recorded at a step size of 0.3–0.4 μm at various magnifications to allow for sufficient number of grains per field of view. All post-processing of maps, the grain size analysis and misorientation angle calculation, were performed in HKL Channel5 software.

Noise reduction was performed on EBSD patterns, with removal of wild spikes and zero solutions. Wild spikes are considered single pixels, that have a completely different orientation than the surrounding pixels, if these share a common orientation. Zero solutions are reduced if more than 4 neighboring grains are indexed, then removal and replacement with the neighbor average will occur. Grain reconstruction was based on a 5° minimum grain threshold angle, and minimum grain sizes of 20–200 pixels, depending on the grain size; the thresholds for low angle grain boundaries (LAGB) ≥ 5°, and ≥10° for high angle grain boundaries (HAGB), respectively. CSL-type boundaries were excluded from the grain size measurements, using 5° maximum angular deviation for the detection. This is a tighter range than suggested by the commonly employed Brandon’s criterion [13], but follows approaches suggested by e.g. Randle [14]. Determination of the fraction of recrystallized grains and the DRX grain size was done using the well-known grain orientation spread (GOS) approach [15–17], which gives the averaged difference of the misorientations between each pixel of a grain and the grain’s mean misorientation. The GOS of a recrystallized grain will be much lower than for deformed or substructured grains, the threshold for the present alloys was between 2° and 3°. Grain size determination was performed using the equivalent circle...
diameter (ECD) based on statistically significant datasets of many hundreds of grains per field-of-view.

Modeling of the stable phases in equilibrium was performed using ThermoCalc 2015a with the TCNi7.1 database, the stable phase diagrams for all three alloys can be found in the appendix, including a scaled view of the temperature range of interest. For the temperature range used in the mechanical testing, all alloys are predicted to have some amounts of secondary phases stable in equilibrium, except for Haynes 214, which only shows the γ'-matrix. Both Haynes 230 and Inconel 740H are predicted to form primary carbides from the melt, stable to temperatures below 1000 °C. For Inconel 740H, this is a (Nb,Ti)C-type carbide, while for Haynes 230 it is a tungsten-rich M6C-type carbide. In addition, Haynes 230 is predicted to form a M23C6-type carbide, also with FCCL12-type structure, stable from 1160 °C to below 1000 °C. In addition, due its aging treatment at 800 °C, Inconel 740H is expected to have initially some γ'-phase present, likely to be metastable due to the projected γ'-dissolution temperature of 995 °C.

Acknowledgments

The authors would like to thank Dr. George Young at Dominion Engineering in Reston, VA, Mr. Evgueni Poliak at Arcelor Mittal R&D in East Chicago, IN and Dr. Martin Detrois at NETL in Albany, OR, for the valuable help in proofreading and the technical discussions about this paper. Thanks to Brian Baker from Special Metals for technical assistance and important background information.

Thanks to Tony Chen and Greg Baty at the Center for Electron Microscopy and Nanofabrication at Portland State University for assistance with SEM work.

Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References