

4 **Experimental Methodology for Studying Strain Heterogeneity**  
5 **with Microstructural Data from High Temperature Deformation**8 **Garrett J. Pataky · Huseyin Sehitoglu**

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12 **Abstract** An experimental methodology has been developed  
13 to study materials deformed at high temperatures using high  
14 resolution *ex situ* digital image correlation (DIC). This study is  
15 an advancement of techniques that have used sub-grain level  
16 strain measurements and linked them to microstructural data  
17 obtained with electron back-scatter diffraction (EBSD). The  
18 approach utilized air blasting particles that were capable of  
19 remaining unchanged onto a sample surface and loading the  
20 sample at elevated temperatures in vacuum to prevent oxidation.  
21 Two tensile experiments were performed at temperatures  
22 of 700 and 800 °C on Haynes 230 (a nickel-based superalloy)  
23 using DIC at high magnifications in order to study the hetero-  
24 geneous strain fields. Overlaying the strain fields and micro-  
25 structure data, areas of high strain were studied at a grain  
26 boundary, triple junction of boundaries, and at a slip band.  
27 The strain evolution at these areas was compared to the mean  
28 strain accumulation. This study represents an advancement of  
29 the usefulness of high resolution DIC by expanding the load-  
30 ing conditions that can be studied and provided experimental  
31 results to prove the feasibility.

32 **Keywords** Digital image correlation · Electron  
33 backscattering · Microstructure · High temperature ·  
34 Experimental procedure · Deformation

**Introduction** 35

To assist the development of physically based models, there is 36  
a need to experimentally study strain heterogeneities at the 37  
sub-grain level [1–3]. There have been continual develop- 38  
ments to increase the resolution of measurement techniques 39  
in order to meet this demand. Many studies have successfully 40  
linked strains to the microstructure using electron back-scatter 41  
diffraction (EBSD). Currently, there are few high resolution 42  
experimental studies performed on materials deformed at high 43  
temperatures [4, 5]. The present study will focus on describing 44  
the experimental methodology developed in order to study a 45  
nickel-based superalloy at high temperatures using sub-grain 46  
measurements from digital image correlation (DIC) along 47  
with preliminary results of two tensile tests. 48

Two experimental, optical methods have emerged as being 49  
useful for studying strain heterogeneities. The first method is a 50  
grid method used by several researchers [6–8]. An explanation 51  
of this displacement measurement technique can be found in 52  
[9]. The problem with this measurement technique is that 53  
localized high gradients have been found to be difficult to 54  
detect [6]. The current study aims to observe the strain het- 55  
erogeneities across grain boundaries, twin boundaries, and 56  
carbides, and thus the ability to measure these high gradients 57  
is extremely important. 58

The second method to experimentally measure dis- 59  
placements is digital image correlation (DIC). Instead of 60  
tracking a grid during deformation, DIC uses the light 61  
intensity of each pixel in the full-field, separated into 62  
unique boxes called subsets, to quantitatively measure 63  
displacements. There is no inherent length scale for DIC 64  
and a higher resolution measurement is produced by 65  
increasing the magnification of the images captured. 66  
This allows for sub-grain level measurements without 67  
a large grain size. A more detailed description of the 68  
DIC technique is given in [10]. 69

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70 Two approaches to the experiments are possible. *In situ*  
 71 provides measurements during loading without having to  
 72 remove the specimen in order to capture images. This has  
 73 been widely used by researchers to study, for example, fatigue  
 74 crack growth [11, 12] and monotonic loading [13]. The issue  
 75 with the *in-situ* approach is that it generally must be performed  
 76 at lower magnifications in order to avoid being affected by  
 77 vibrations causing blurring of the images. In order to circum-  
 78 vent this issue, special load frames have been used in con-  
 79 junction with scanning electron microscopes (SEM) or optical  
 80 microscopes [8, 14, 15]. There is a limitation to this though; it  
 81 is difficult to produce high temperature experiments without  
 82 damaging the equipment and specimen geometries are limit-  
 83 ed. The other approach, *ex situ*, is not limited by the experi-  
 84 mental setup or environment, including high temperature,  
 85 since the sample is removed from the testing apparatus before  
 86 imaging. This method can be performed either optically or in  
 87 the SEM. Optical microscopes are limited in their depth of  
 88 focus, which is detrimental in the case of out of plane dis-  
 89 placements. Using an SEM, higher magnifications can be  
 90 achieved, but this is not without its own issues. Drift during  
 91 image capture and background noise affecting the pattern are  
 92 two of the major problems [16]. An optical microscope was  
 93 used in this study in order to circumvent these drawbacks  
 94 since out of plane displacements will be small.

95 The present study will introduce a high resolution DIC *ex*  
 96 *situ* experimental methodology in for studying materials de-  
 97 formed at high temperatures. The strain fields of a  
 98 predetermined area will be measured and related to the micro-  
 99 structure using data found from EBSD. The procedure used is  
 100 an advancement of the one developed by Carroll et al. during a  
 101 similar study performed on materials at room temperatures  
 102 [17]. The results from Haynes 230, a nickel-based superalloy,  
 103 deformed at 700 °C and 800 °C under monotonic loading will  
 104 be shown using this technique. The abilities developed by this  
 105 study will be a clear advancement for studying strain hetero-  
 106 geneity in materials in a high temperature environment.

107 **Experimental Procedure**

108 The following section will outline the experimental procedure  
 109 used to perform *ex situ* high resolution DIC. This methodol-  
 110 ogy has been utilized to study a nickel-based superalloy at  
 111 700 °C and 800 °C. Every step of the experiment will be  
 112 described including the sample preparation, experimental set-  
 113 up, deformation analysis, and alignment of the DIC strain  
 114 fields to the microstructure data obtained using EBSD.

115 **Sample Preparation**

116 In order to perform EBSD and get information about the  
 117 microstructure, the samples must be polished to a mirror

finish. The specimens used during this study were “dog-bone” 118  
 shaped with a gauge length of 25.00 mm, width of 4.00 mm, 119  
 and a thickness of 2.38 mm. This procedure is not limited by 120  
 the specimen shape or size, thus any specimen can be used. 121  
 The samples were mechanically polished with abrasive grind- 122  
 ing paper up to a grit of P4000. The sample was then polished 123  
 using 0.3 μm silica powder and finalized with a vibropolisher. 124

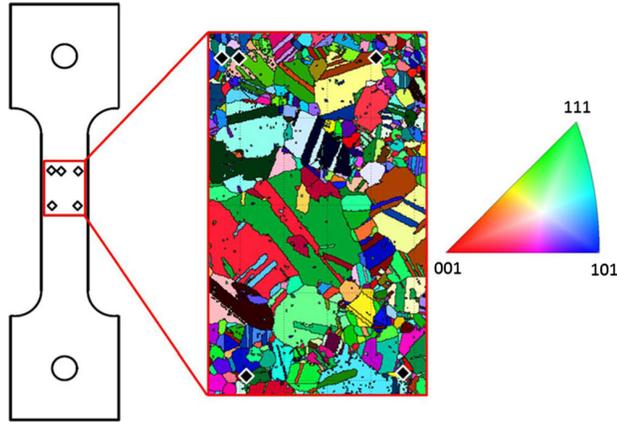
125 Before the microstructure was characterized, the area of 125  
 interest on the sample surface needed to be marked. A discus- 126  
 sion of the requirements for these marks is given by Carroll 127  
 et al. with Vickers indentation marks being determined as 128  
 ideal [17]. The main requirements of these markings were that 129  
 they need to last throughout the entirety of the experiment so 130  
 all data sets can be linked, and they must be small enough to 131  
 align with the microstructure. Five Vickers marks were used to 132  
 mark off a rectangular area of 1.05 mm by 0.525 mm. The 133  
 fifth mark was made next to one of the corners to discern the 134  
 alignment of the area. EBSD was performed in a JEOL 135  
 7,000 F SEM with measurement spacing of 1.0 μm. Due to 136  
 the size of the area of interest, multiple EBSD scans were 137  
 made. These were aligned and joined with the CHANNEL 5 138  
 software package. Figure 1 shows a schematic of the sample 139  
 used, the location of the area of interest, and the EBSD scan of 140  
 the area. 141

142 The next step of the experiment was to speckle the sample 142  
 surface in order to use DIC. This was a major area of concern 143  
 since anything on the surface can be destroyed by the elevated 144  
 temperatures thus destroying the speckle pattern. There are a 145  
 couple requirements that must be met. First, the speckles of 146  
 the pattern must be small enough in order to produce sub-grain 147  
 quality measurements. The material deposited on the surface 148  
 must be able to withstand the temperature of the experiment as 149  
 well. Depositing particles onto the surface using compressed 150  
 air was identified as the best method, as first used by 151  
 Jonnalagadda et al. [18]. This is due to the consistency a 152  
 pattern can be made without worrying about having random 153  
 large speckles, as seen with spraying paint, and the large range 154  
 of materials available to deposit. Two types of particles were 155  
 tested, 1 μm diameter Si and 0.3 μm alumina. Once deposited 156  
 on the surface, both types of particles provided good DIC 157  
 speckle patterns, Si shown in Fig. 2a and alumina shown in 158  
 Fig. 2b. After heating to 800 °C in vacuum, cooling, and 159  
 removing, it was immediately obvious that the Si would not 160  
 suitable for this application (Fig. 2c) but the alumina remained 161  
 unchanged (Fig. 2d). This example expounded the second 162  
 requirement for the speckle material. 163

164 **Experimental Procedure**

165 The procedure outlined is for performing 2-D DIC for a 165  
 material deformed under high temperature conditions. A vac- 166  
 uum system is employed in order to prevent oxidation which 167  
 would destroy the DIC speckle pattern. Since the 168





**Fig. 1** A schematic of the “dog-bone” specimen with the location of the area studied defined by the Vickers indentation marks. The EBSD scan results showing grain orientations with the Vickers marks accentuated

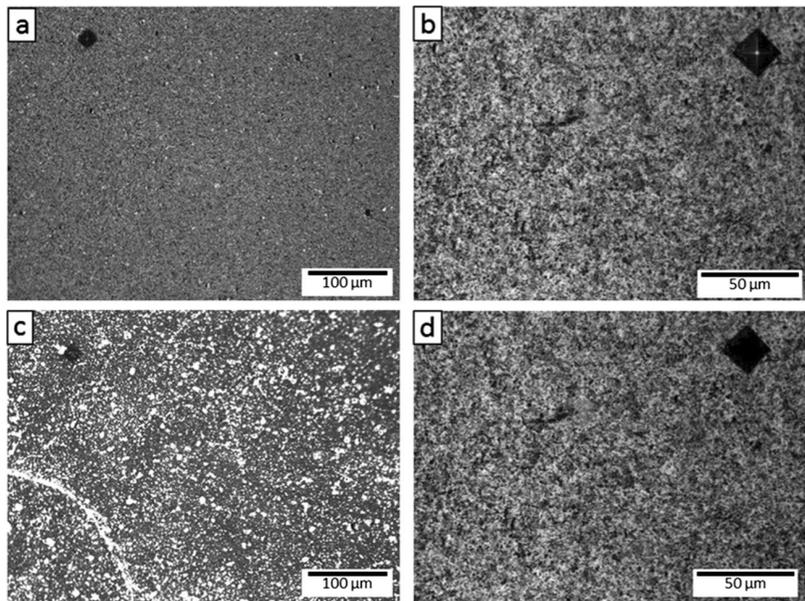
169 heterogeneity of the strain field at the microstructural level is  
 170 desired, a microscope has been used to capture the images.  
 171 This allows for higher spatial resolution compared to previous  
 172 high temperature DIC studies [13, 19]. The following section  
 173 outlines the equipment used and the procedure performed.

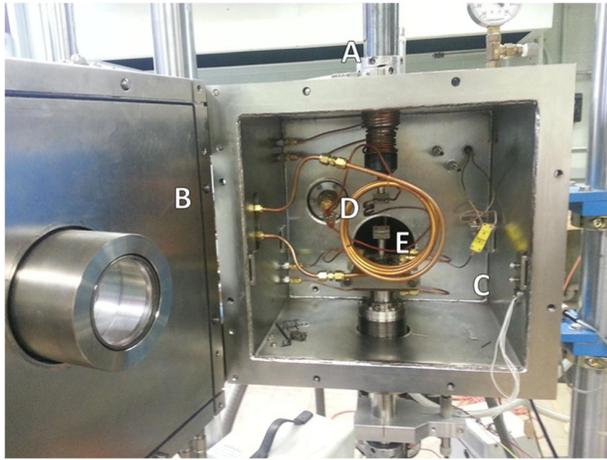
174 An initial set of images of the sample surface with the  
 175 alumina speckle pattern (Section 2.1) was taken as the DIC  
 176 reference images. Since the DIC measurement resolution is  
 177 based on the imaging magnification, consideration must be  
 178 made to choose a magnification that will elucidate the desired  
 179 sub-grain level measurements. A limitation to using higher  
 180 magnifications was the reduced area that each image captured.  
 181 In order to circumvent this issue, multiple images must be  
 182 taken in order to capture the entire area of interest. In this

183 study, an optical microscope and two magnifications were  
 184 used with an IMI 202FT digital camera that had a resolution  
 185 of 1,600 pix by 1,200 pix. The lower magnification used was  
 186 10× which corresponded to a scale of 0.437 μm/pix and a  
 187 higher magnification of 25× which corresponded to a scale of  
 188 0.175 μm/pix. 3 images at the magnification of 10× and 27  
 189 images at the magnification of 25× were required to cover the  
 190 entire area of interest. The explanation on how to handle the  
 191 image array will be covered in Section 2.3.

192 After the set of references images was taken, the sample  
 193 was ready to be prepared for the experiment. A vacuum or  
 194 inert environment is necessary to safeguard against uncontrol-  
 195 lable pattern changes once the sample is taken to temperature  
 196 during testing. A vacuum chamber was utilized during this  
 197 study to protect the sample surface from oxidation. A picture  
 198 showing the vacuum chamber and load frame used during this  
 199 study is given in Fig. 3. The specimen had a type-K thermo-  
 200 couple spot welded onto the middle of the back once placed  
 201 into the load frame. The chamber was then sealed and pulled  
 202 into a vacuum of 10<sup>-6</sup> Torr. Once this level was reached,  
 203 which typically took pumping the system overnight, the sam-  
 204 ple was heated using induction heating at a rate of 75 °C per  
 205 minute. Once the specimen had reached the desired tempera-  
 206 ture, 700 °C or 800 °C for this study, the temperature was kept  
 207 to a tolerance of ±3 °C for the duration of the deformation step  
 208 using a temperature controller connected to the induction  
 209 heater. The specimen was then loaded with a servo-hydraulic  
 210 load frame. After the desired amount of deformation, mono-  
 211 tonic tension at a strain rate of 10<sup>-4</sup> s<sup>-1</sup> in this study, was  
 212 reached, the system was cooled down to room temperature  
 213 before returning the chamber to atmospheric pressure. This  
 214 was to guarantee that there would be no avoidable surface

**Fig. 2** Speckle patterns of air blasted (a) Si particles (10× magnification) and (b) alumina particles (25× magnifications) before heating taken using the optical microscope. (c) The pattern with the Si particles changed drastically after being heated to 800 °C while the (d) alumina particle pattern remained consistent with the unheated pattern





**Fig. 3** A picture of the experimental setup showing: (a) load frame, (b) vacuum chamber, (c) type-K thermocouple, (d) induction heating coils, and (e) grips where specimen was placed. The copper coiling is to provide cooling during the experiments at elevated temperatures

215 changes. The sample was then removed from the chamber and  
 216 the thermocouple taken off. Images of the deformed surface  
 217 were then captured with the optical microscope in the same  
 218 method that the reference images were captured. The Vickers  
 219 markers made visually identifying the area of interest easy. It  
 220 is pertinent to capture the images in the same pattern as the  
 221 reference images to avoid any unnecessary post-processing  
 222 changes. Once the imaging was completed, the procedure of  
 223 attaching the thermocouple, pulling vacuum, heating the sam-  
 224 ple, and then deforming was repeated. For the experiment  
 225 performed at 800 °C two deformation steps were made and  
 226 for the 700 °C experiment ten deformation steps were made.

227 **Analysis**

228 After deformation and image capture, the next step was to  
 229 analyze the data using DIC. Since this method is an *ex situ*  
 230 technique, the strain fields found will be the residual plastic  
 231 strains. There will be two methods discussed for completing  
 232 this since multiple images were required to cover the area of  
 233 interest: stitching the images and stitching the DIC results.  
 234 Both methods were used during this study. The first technique,  
 235 stitching the images and then correlating, was used for the  
 236 images magnified at 25× in the 800 °C experiment and the  
 237 images magnified at 10× in the 700 °C experiment. The  
 238 stitching of the images was performed with a stitching plug-  
 239 in available in the open-source, image processing package  
 240 FIJI [20]. The plug-in uses a Fourier Shift Theorem that  
 241 computes all possible translations in order to find the optimal  
 242 overlapping. Each set of images, reference and deformed, was  
 243 stitched yielding a fused image. The deformed images were  
 244 then correlated to the reference images using commercial DIC  
 245 software. There is a couple drawbacks to this technique. The

246 25× magnification stitched images were very large, over 100  
 247 megabytes, and this exceeded the limit of the software to  
 248 correlate, and the stitching seams have been found to produce  
 249 artificial strains [17]. It is important to note that both times the  
 250 25× magnification was utilized, the correlating parameters  
 251 were kept the same.

252 The second method to handle the image array is to perform  
 253 DIC on each individual image and then combine those results  
 254 as developed by Carroll et al. [17]. This approach was used for  
 255 each set of the 27 images taken at the 25× magnification  
 256 during the 700 °C experiment. Each of these images was  
 257 overlapping by 40–50 % to create enough overlapping data  
 258 to match up the DIC results sufficiently. This produced 11 sets  
 259 of 27 strain fields (the reference and 10 deformed sets).  
 260 Deformation caused each of the strain fields to be at a slightly  
 261 different location. To get a reference location for the strain  
 262 fields, the reference images were stitched together using FIJI,  
 263 as previously discussed, providing their original locations.  
 264 Subsequently the strain fields were interpolated into an evenly  
 265 spaced grid, mimicking a DIC subset grid, to produce the  
 266 entire full-field strain field using these original locations.  
 267 The strains were stitched to alleviate the issue of seams  
 268 producing errors since this stitching occurred post-  
 269 correlation. This reduced the error down to interpolation in-  
 270 stead of artificial strains.

271 The final step was to align the DIC strain fields and the  
 272 microstructure data from the EBSD. This was done using the  
 273 Vickers marks made on the surface of the specimen at the start  
 274 of the experiment. The Vickers marks are difficult to deter-  
 275 mine their exact location and size from the strain fields, so the  
 276 first step was to overlay each of the strain fields onto the  
 277 stitched reference image since these locations are known. This  
 278 was possible with software that allows layers, such as Adobe  
 279 Photoshop or the open-source software GIMP. Then the  
 280 EBSD data can be overlaid onto the stitched reference image  
 281 by aligning the Vickers marks. This required some manipula-  
 282 tion to the EBSD data since it was skewed and rotated. After  
 283 this was completed, the stitched reference image was removed  
 284 leaving the strain fields and microstructural data aligned. It  
 285 was then possible to determine how the grain boundaries, twin  
 286 boundaries, and carbides affected the strain heterogeneity.  
 287 Each of the boundary types are known as well so it is possible  
 288 to do statistical analysis such as in references [21, 22].  
 289

290 **Results and Discussion**

291 The following section will present the results from the mono-  
 292 tonic tension experiments performed at 700 and 800 °C and  
 293 analyzed using the high resolution *ex situ* DIC technique  
 294 presented in this paper. The 800 °C experiment produced large  
 295 scale cracking after two deformation steps. Subsequently, the



296 700 °C was performed and ten deformation steps were able to  
297 be produced.

298 Measurement Error

299 When performing DIC, one of the major concerns is the  
300 amount of measurement error present. It was concluded in  
301 an earlier study that the image stitching technique provided a  
302 larger amount of error than the DIC stitching technique [17].  
303 The following will cover the error determined for this study  
304 based on the  $\epsilon_{yy}$  strain since this was the component presented  
305 in this study.

306 The first step in reducing the error was to ensure that the  
307 specimen was inserted into the microscope in a consistent  
308 manner. A custom slide was designed in order to hold the  
309 specimen in the same spot and orientation each time it was  
310 placed on the microscope. The slide was designed so it could  
311 be attached to the microscope easily and was rigidly attached.

312 The error analysis is based on the 700 °C experiment since  
313 the out of plane displacements due to cracking were not  
314 observed. Two sets of reference images at 10× magnification  
315 for the 700 °C experiment were captured for the error analysis.  
316 This was done by taking one complete set of images, remov-  
317 ing the specimen from the microscope, and then replacing the  
318 specimen and capturing the second set of images. The micro-  
319 scope settings (brightness, shutter speed, sharpness, etc.) were  
320 kept the same. This provided a base set of two reference,  
321 stitched images in which the average strains throughout the  
322 entire experiment could be compared. Each stitched deforma-  
323 tion image was correlated to the same area of interest for both  
324 of the reference images. Table 1 gives the comparison between  
325 the mean  $\epsilon_{yy}$  strains found. The results showed a maximum  
326 background error of 0.0023 %. Although impressive, the local  
327 error is an important number to consider since this study  
328 concentrated on the strain heterogeneity. To find the local  
329 error, the entire field was analyzed for the highest strain  
330 difference between the two identical fields. The two reference  
331 images were correlated to each other, both being undeformed,  
332 and the maximum local error was found to be orders of  
333 magnitude higher at 0.11 %. The local error was determined  
334 again using the tenth deformation step by finding the maxi-  
335 mum difference between correlations when correlating this  
336 image array to both of the undeformed, reference images.  
337 The magnitude of error measured was to be 0.20 %. It must  
338 be noted that this was determined at a region with strain over  
339 2 %.

340 A similar error analysis was performed with the 25× mag-  
341 nification images. This procedure was performed with the  
342 DIC data stitching method. When comparing two undeformed  
343 sets of images, the mean  $\epsilon_{yy}$  strain error was found to be  
344 0.0096 %. This was on the order found with the 10× magni-  
345 fication. The local error was found to have increased to  
346 0.34 %. When taking into consideration the strains found at

**Table 1** A comparison of the DIC mean  $\epsilon_{yy}$  strain values over the entire area of interest correlated to each of the two 10× magnification reference images

	Reference 1	Reference 2	Difference	
Step 1	0.022 %	0.024 %	0.0018 %	t1.3
Step 2	0.295 %	0.297 %	0.0013 %	t1.4
Step 3	1.061 %	1.062 %	0.0012 %	t1.5
Step 4	1.216 %	1.218 %	0.0020 %	t1.6
Step 5	1.369 %	1.370 %	0.0015 %	t1.7
Step 6	1.586 %	1.587 %	0.0016 %	t1.8
Step 7	1.872 %	1.874 %	0.0016 %	t1.9
Step 8	1.961 %	1.963 %	0.0020 %	t1.10
Step 9	2.511 %	2.513 %	0.0022 %	t1.11
Step 10	3.094 %	3.096 %	0.0023 %	t1.12

the areas of concentration studied in Section 3.3, the result is a  
low amount of error.

Material

The material used during this study was the nickel-based  
superalloy Haynes 230. Haynes 230 is a solid-solution  
strengthened alloy that was developed for high temperature  
applications and thus has additions of tungsten, chromium,  
and molybdenum. The grains were found to be equiaxed with  
grain sizes ranging from 30  $\mu\text{m}$  to 250  $\mu\text{m}$ . The EBSD data  
showed a large amount of annealing twins and two-thirds of  
the boundaries were observed to be  $\Sigma 3$  boundaries following  
the coincident lattice notation (CSL). An example of the  
microstructure was given in Fig. 1.

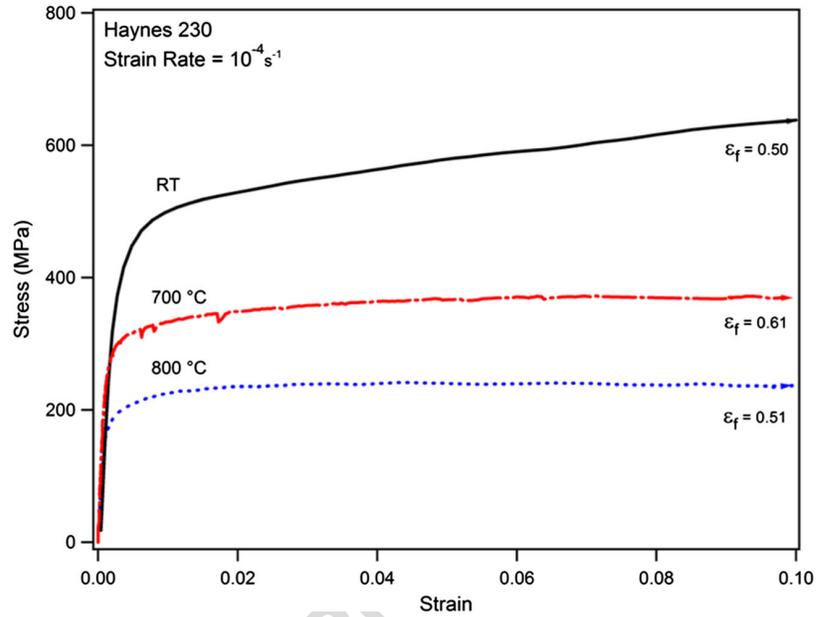
The results of tensile tests performed at room temperature,  
700, and 800 °C are given in Fig. 4. All three experiments  
were run at a strain rate of  $10^{-4} \text{ s}^{-1}$ . The yield stress for each  
temperature was found using a 0.2 % offset. This value was  
found to be 415 MPa at room temperature, 302 MPa at  
700 °C, and 197 MPa at 800 °C. Hardening was observed in  
the room temperature results and initially in the 700 °C results,  
while the 800 °C results showed softening. Serrations in the  
700 °C stress–strain curve were attributed to the Portevin-Le  
Chatelier effect, but not in the 800 °C stress–strain curve. This  
has also been found in other similar nickel-based superalloys,  
such as Hastelloy X [23].

800 °C Tensile Test

Two steps of deformation were made at 800 °C using the  
experimental technique outlined in Section 2. A 51 pix by 51  
pix (8.9  $\mu\text{m}$  by 8.9  $\mu\text{m}$ ) subset size with a step of 5 pix  
(0.875  $\mu\text{m}$ ) was used for the DIC analysis. The first step  
was measured at a mean  $\epsilon_{yy}$  strain of 0.8 %, and the second  
at 5.5 %. During the second step, many cracks formed on the



**Fig. 4** The stress–strain response of Haynes 230 at room temperature (RT), 700 °C, and 800 °C

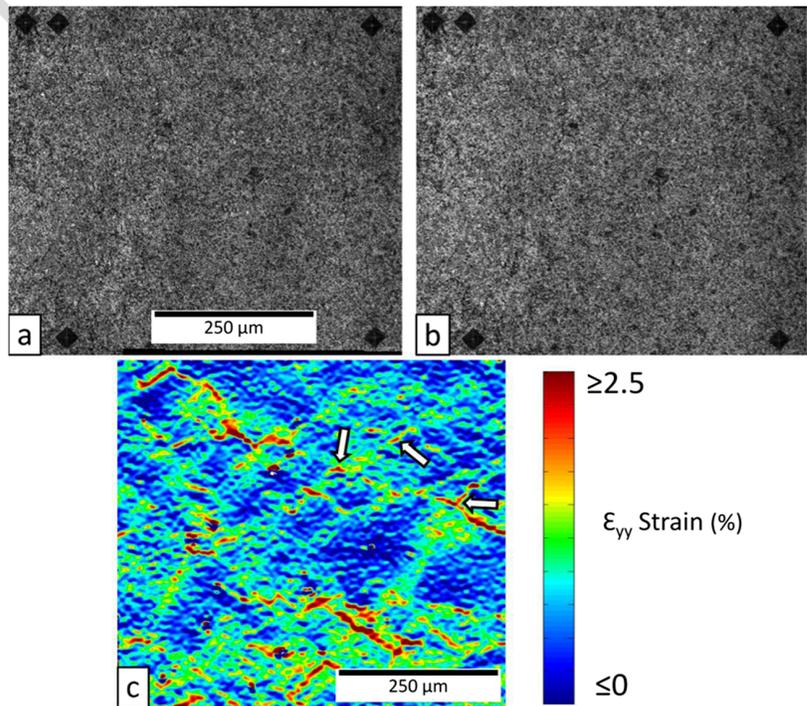


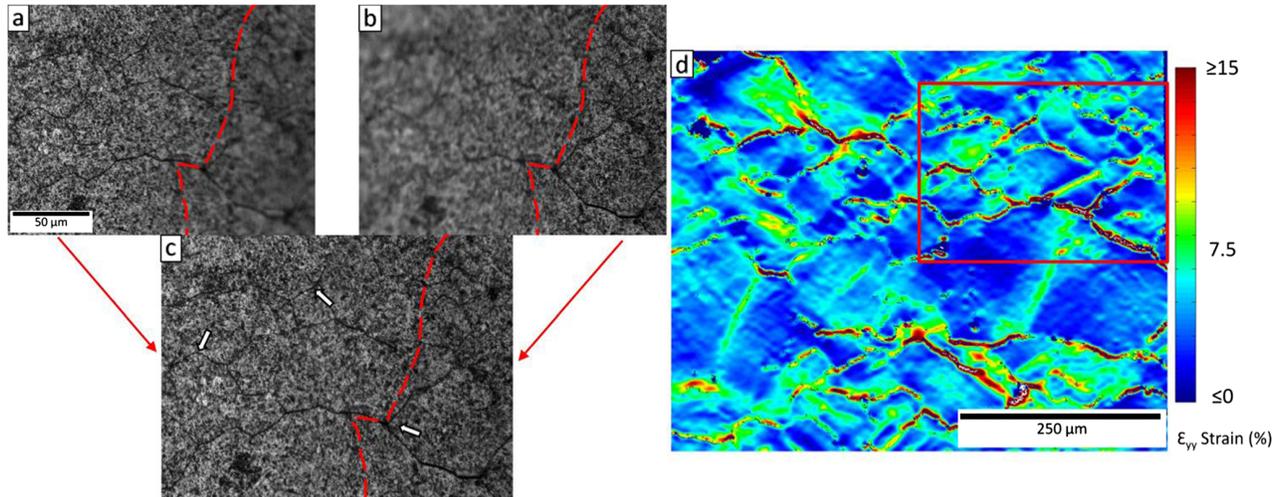
379 Haynes 230 sample. These showed up as very high strains  
 380 (over 15 %) in DIC and thus the 5.5 % of mean strain was  
 381 considered to be an overestimation. The stitched reference  
 382 image, stitched first step image, and the DIC results are given  
 383 in Fig. 5a–c respectively. It is clear that even with the mean  
 384 strain under 1 %, there was a heterogeneous strain field  
 385 present. Areas with strains over 2.5 % occurred throughout.

386 Due to the crack formation on the Haynes 230 sample after  
 387 the second deformation step, the sample surface rose in certain

388 areas which made it impossible to capture the complete area of  
 389 interest while still in focus. This phenomenon was not present  
 390 in the entire area of interest and the change in focus followed  
 391 the crack paths. An example of one section is given in Fig. 6.  
 392 In Fig. 6a and b, the red dotted line depicts the divide between  
 393 the focuses. There was a simple procedure to solve this dilemma.  
 394 Capturing images at different focuses (Fig. 6a and b)  
 395 and then combining them in software that allows layers, such  
 396 as the earlier mentioned Adobe Photoshop or GIMP, provided  
 397

**Fig. 5** Images from the optical microscope showing the 0.6 mm by 0.6 mm area and speckle pattern of the sample tested at 800 °C (a) undeformed and (b) after the first step of heating and deformation at a magnification of 25× with the Vickers indentation marks visible. (c) The  $\epsilon_{yy}$  strain field found using DIC. The white arrows in (c) show high strained areas that crack in the next deformation step (shown in Fig. 6c)





**Fig. 6** (a) and (b) Images taken with the optical microscope depicting the lack of focus in certain areas of each image. (c) The combined image created using the images in (a) and (b) and eliminating the out of focus regions. (d) The  $\epsilon_{yy}$  strain field found using DIC with cracks on the sample showing strain magnitudes of 15 % or greater. The red box is the region correlated using the image in (c). The white arrows in (c) show cracked regions that were the highly strained areas in the previous deformation step

397 the base. By eliminating the areas out of focus on the top layer,  
 398 an image that has full focus is obtained, as shown in Fig. 6c.  
 399 Once this was completed, the DIC correlation was performed  
 400 and the axial strain results are given in Fig. 6d. The red box  
 401 identifies the area from Fig. 6c, showing the success of this  
 402 technique. It must be stated that since this is a manual manip-  
 403 ulation technique, this procedure should be limited to prevent  
 404 the introduction of error. Comparing Figs. 5c and 6c, there  
 405 was a clear correlation between the areas of high strain con-  
 406 centration in the first step and the eventual crack locations in  
 407 the second step. Three of the high strain regions which  
 408 cracked are highlighted with white arrows. A majority of these  
 409 cracks were along grain boundaries.

410 700 °C Tensile Test

411 This tensile experiment was performed at 700 °C and included  
 412 10 deformation steps. Two magnifications were used. The  
 413 images taken at a magnification of 10× were analyzed using  
 414 the image stitching technique, while the images taken at 25×  
 415 magnification were analyzed using the DIC data stitching  
 416 technique. The DIC analysis for the 10× magnification was  
 417 performed with a 31 pix by 31 pix (13.55 μm by 13.55 μm)  
 418 subset size with a step of 5 pix (2.185 μm) and the 25×  
 419 magnification analysis was performed with the same subset  
 420 as the 800 °C test, 51 pix by 51 pix (8.9 μm by 8.9 μm) subset  
 421 size with a step size of 5 pix (0.875 μm). The reason for using  
 422 the lower magnification was to be able to quickly determine  
 423 the mean strains. This only required stitching 3 images togeth-  
 424 er per deformation and performing the DIC analysis. The  
 425 higher magnification results were used in conjunction with  
 426 the microstructural data to elucidate information about its  
 427 effects on the strain heterogeneity. The stress–strain curve

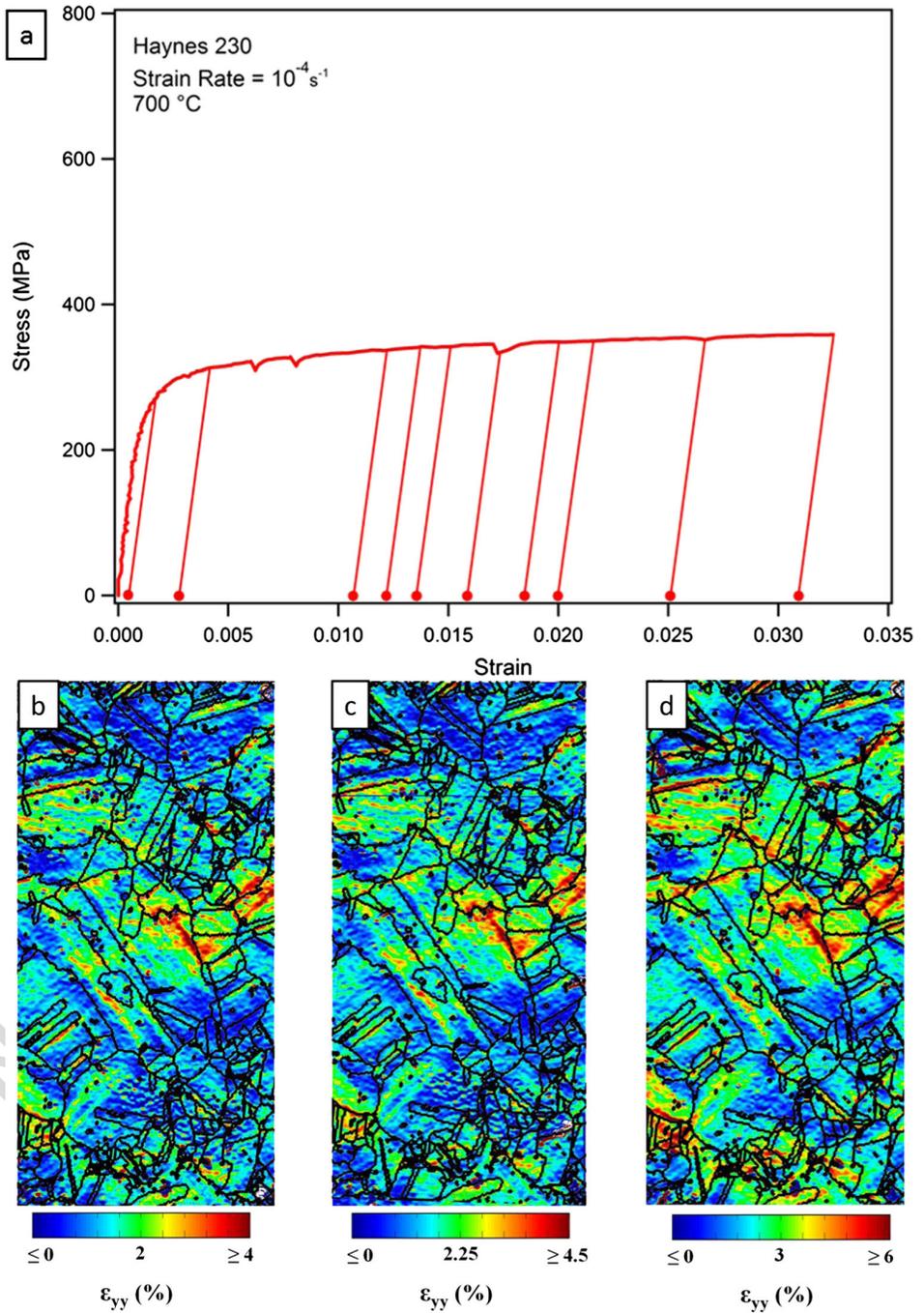
with each of the deformation steps marked is given in  
 Fig. 7a. The deformation steps in terms of the mean  $\epsilon_{yy}$  strain  
 were as follows: 0.02, 0.30, 1.06, 1.22, 1.37, 1.59, 1.87, 1.96,  
 2.51, and 3.09 %. The three DIC  $\epsilon_{yy}$  strain fields, Fig. 7b–d,  
 are the higher magnification results from the 1.22, 1.59, and  
 2.51 % steps respectively.

The increasing strains are obvious in Fig. 7 when taking  
 into account the scales. The use of both DIC strain fields and  
 microstructure data from EBSD provided insight into the main  
 areas of strain concentration during the deformation.

To quantitatively find results, the strains were analyzed  
 globally. Figure 8 presents these results. In Fig. 8a, a histo-  
 gram depicts the changing strain values for four of the deforma-  
 tion steps. It is observed that there is a clear shift from a  
 peak of concentrated values for Step 2 (mean  $\epsilon_{yy}$  strain of  
 0.32 %) to a broad extended distribution of strains in Step 9  
 (mean  $\epsilon_{yy}$  strain of 2.53 %). From this result and the hetero-  
 geneity shown in Fig. 7, it can be interpreted that some areas  
 are deforming at a higher rate than others while certain areas  
 are not deforming. A clearer picture of this is given in Fig. 8b  
 which illustrates that the maximum strain is increasing at a  
 quicker rate than the mean value. It is also noted that the  
 minimum strain value is consistent throughout the entire ex-  
 periment. The standard deviation of the  $\epsilon_{yy}$  strain field, plotted  
 in Fig. 8c, quantitatively shows the increase of heterogeneity  
 perceived from the other figures.

Since the EBSD data is rich with information about grain  
 orientations and grain boundary types, it is possible to com-  
 bine this with the DIC results to look at the activated slip  
 systems. The method used can be found in [21]. For brevity  
 since the focus of this paper is about abilities and the high  
 temperature technique, only two slip systems (7 and 8) have  
 been shown. The corresponding slip plane and slip direction

**Fig. 7 (a)** The stress–strain response of Haynes 230 experimented at 700 °C with the 10 steps of deformation indicated when the experiment was halted to image for DIC. The  $\epsilon_{yy}$  strain field of the 1.05 mm by 0.525 mm area studied with the grain and twin boundaries overlaid at mean strains of (a) 1.22 %, (b) 1.59 %, and (c) 2.51 %. Note the scale bar changes for each strain field



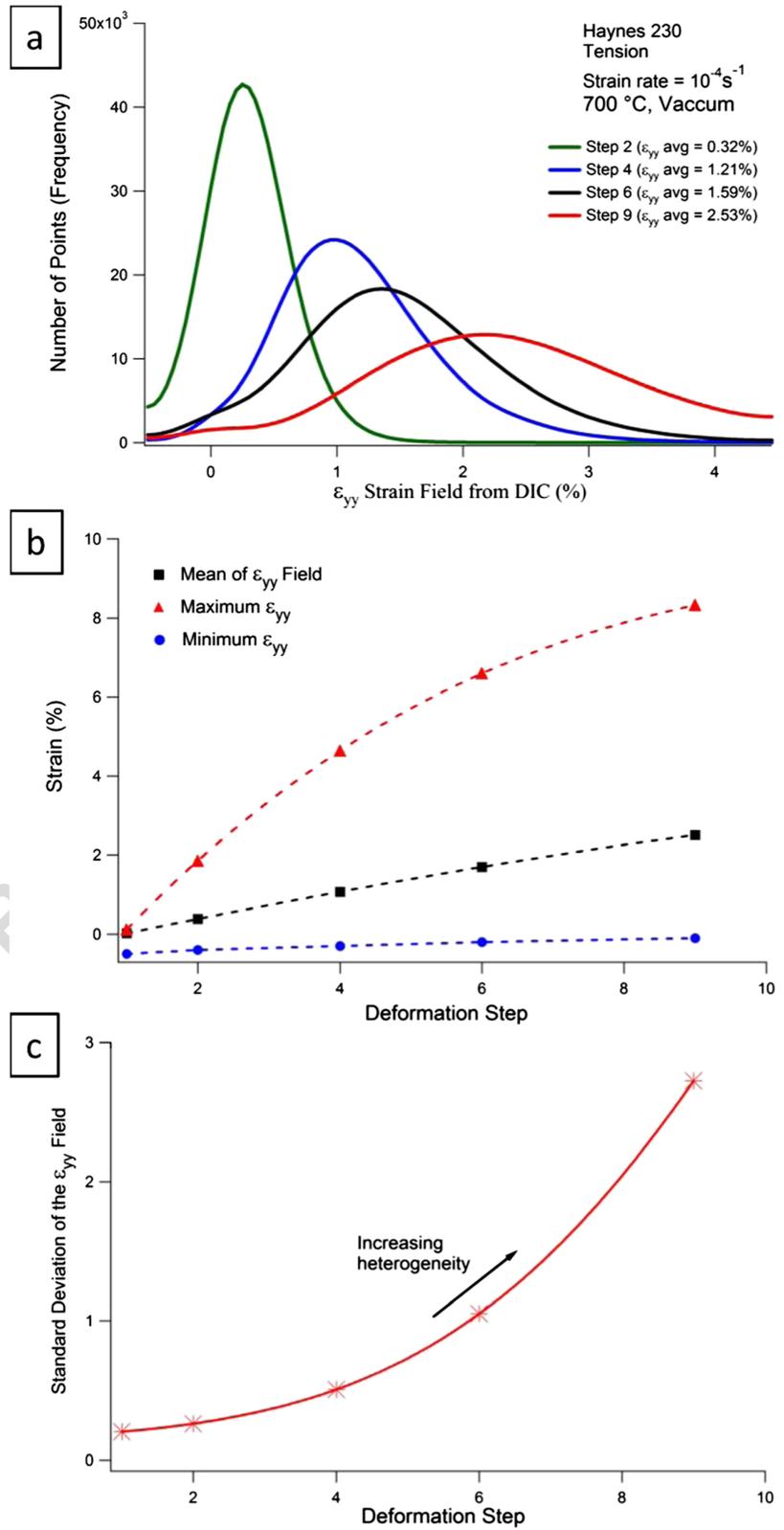
461 for these systems are highlighted in Table 2. Figure 8 (system  
 462 7) and Fig. 9 (system 8) show the results from deformation  
 463 step 6. The dark blue areas (0 % strain) indicate that that  
 464 respective system has not been activated in the grain. The  
 465 areas boxed and magnified show an annealing twin, a  $\Sigma 3$   
 466 boundary using the CSL notation, inside of a grain. The  
 467 Schmid factors for the twin and grain are given in Table 2.  
 468 Figure 8 clearly shows that slip is transmitted through the  
 469 grain and twin boundaries, while in Fig. 9 the  $\Sigma 3$  twin

boundary blocks the slip transmission. This is important for  
 studying crack initiation as this can determine the creation of a  
 grain cluster when slip is transmitted or an area of strain  
 concentration in the case of blocking. Further global analysis  
 of this can be performed, including the relationship between  
 strain and residual Burgers vectors [21].

Another part of the microstructure that must be noted is the  
 carbides that are present in this material. Two types of carbides  
 have been identified in several studies of Haynes 230:  $M_6C$

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**Fig. 8** (a) Histogram of the strain values for four deformation steps showing the increasing strain values. (b) The trends of the minimum, maximum, and mean values of the  $\epsilon_{yy}$  strain field for each deformation step. (c) The standard deviation of the  $\epsilon_{yy}$  strain field showing an increasing heterogeneity in the strain field



**Table 2** Schmid factors for the 12 slip systems of the grain and twin shown in Fig. 9

	Slip system, $\alpha$	Slip plane	Slip direction	Schmid factor (Twin)	Schmid factor (Grain)
t2.3	System 1	(111)	$[\bar{1}0]$	0.33	0.03
t2.4	System 2	(111)	$[\bar{1}0\bar{1}]$	0.40	0.01
t2.5	System 3	(111)	$[01\bar{1}]$	0.07	0.02
t2.6	System 4	$(\bar{1}1)$	$[101]$	0.38	0.40
t2.7	System 5	$(\bar{1}1)$	$[01\bar{1}]$	0.09	0.46
t2.8	System 6	$(\bar{1}1)$	$[110]$	0.47	0.07
t2.9	System 7	$(1\bar{1}\bar{1})$	$[\bar{0}1]$	0.37	0.27
t2.10	System 8	$(1\bar{1}\bar{1})$	$[011]$	0.05	0.36
t2.11	System 9	$(1\bar{1}\bar{1})$	$[110]$	0.33	0.09
t2.12	System 10	$(11\bar{1})$	$[\bar{1}0]$	0.46	0.19
t2.13	System 11	$(11\bar{1})$	$[101]$	0.39	0.11
t2.14	System 12	$(11\bar{1})$	$[011]$	0.07	0.08

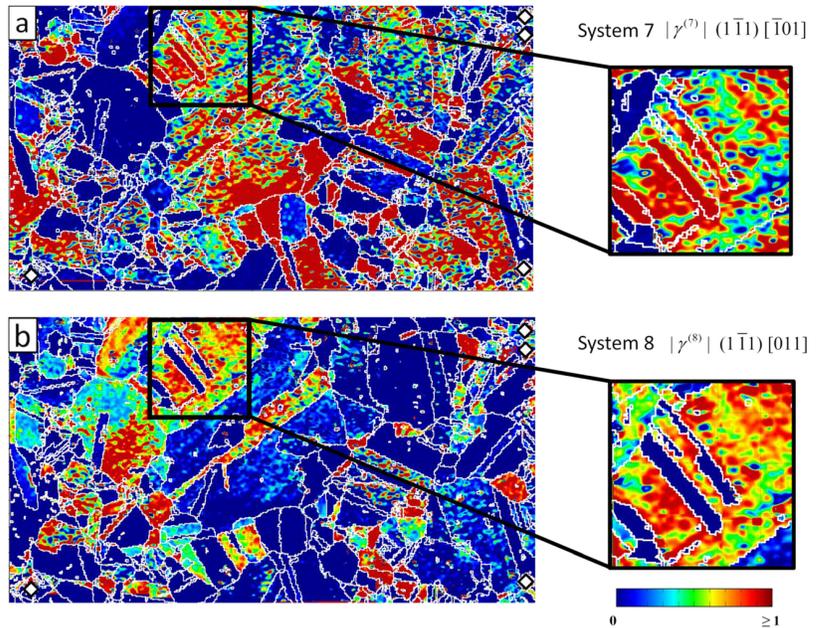
479 and  $M_{23}C_6$  [24, 25]. The M in the  $M_6C$  carbide is assumed to  
 480 be mostly the tungsten while the M in the  $M_{23}C_6$  carbide is  
 481 assumed to be chromium. The DIC analysis begins to show  
 482 failed correlations after the third deformation step at the loca-  
 483 tions that EBSD revealed carbides. This indicates that the  
 484 carbides are much more rigid than the rest of the material  
 485 and play an important role in the deformation at high temper-  
 486 atures. A previous study of creep indicated that carbides at  
 487 grain or twin boundaries in Haynes 230 are likely to pin the  
 488 boundary and prevent sliding reducing creep strain, but even-  
 489 tually leading to a failure of the boundary [25]. This is a  
 490 possible explanation for the large amount of strain concentrat-  
 491 ed at boundaries and the cracking observed in the 800 °C  
 492 experiment. In order to glean all the information provided by  
 493 this experiment, a full analysis of the grain boundary types  
 494 with strain concentrations, active slip systems, and the impact

on strain near carbides is required. This is outside the scope of  
 this study.

### Conclusions

This study presented a new experimental methodology for  
 studying materials at high temperatures with high resolution  
 digital image correlation in conjunction with microstructure  
 data. Previous limitations of the speckle pattern changing due  
 to the conditions in an elevated temperature environment were  
 overcome by using stable alumina particles for a speckle  
 pattern and preventing oxidation by experimenting in vacuum.  
 Using this technique, it is possible to study phenomena such  
 as creep, creep-fatigue, and high temperature monotonic

**Fig. 9** (a) Slip system 7 and (b) slip system 8 shear increments after deformation step 4. The dark blue areas are where the system has not been activated. (a) Slip transmission between a grain and twin is depicted in the magnified box. (b) Blockage of slip from the grain to the twin is shown in the magnified box. See Table 2 for the Schmid factor values



508 tension. DIC was performed on images that were stitched and  
 509 an additional, alternative approach of correlating each indi-  
 510 vidual image and then stitching the DIC results was used.  
 511 Repeatability of this technique was proven by studying  
 512 Haynes 230 under a tensile load at temperatures of 700 °C  
 513 and 800 °C with multiple deformation steps. A method was  
 514 developed to recover the focus of areas with out of plane  
 515 displacement, seen after crack formation in the present study,  
 516 in order to image the surface for DIC analysis. Strain hetero-  
 517 geneity was observed throughout along areas of slip activity,  
 518 grain boundaries, and triple points with strains at these loca-  
 519 tions typically 2 % or greater than the mean strain of the  
 520 region. An error analysis was performed and the errors ob-  
 521 served were small compared to the measured strains. This  
 522 study represents a substantial effort to advance the practicality  
 523 of DIC for studying materials in a variety of environments  
 524 with a high resolution in order to elucidate information about  
 525 the microstructure's effects on strain heterogeneities common-  
 526 ly seen during loading.

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