# HOT-COMPRESSION BEHAVIOR OF Ni-Al-Zr ALLOYS

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**Abstract**

Hot-compression tests of the alloys Ni-Al-Zr were performed. The tests were performed on the equipment Gleeble 3800 at 800 °C. The $Ni_3Al$ based alloys with different content of zirconium were tested in as-cast and directionally solidified state. Solidified samples were prepared from castings by Bridgman's method. The alloys exhibit good values of yield strength and maximal achieved stress for this type of material. The alloys in directed state have better values of yield strength then as-cast alloys. The structure of alloys depends on the aluminum content. The alloys with 24 at.% Al are formed by phases $Ni_3Al$ and Zr-rich phase $Ni_5Zr$. The hypostoichiometric alloys are formed by phases $Ni_3Al$, (Ni) and Zr-rich phase $Ni_5Zr$.

**Keywords:** Compression tests, high temperature characteristics, Ni-Al-Zr Alloys, Bridgman's method

## 1. INTRODUCTION

The $Ni_3Al$ based alloys are modern engineering material. They are used both in cast form with heat treatment, and also after directional solidification for engine turbine blades, for example as part of composite materials. Nowadays it is possible to produce even thin foils and strips for high tech devices. $Ni_3Al$-based alloys are used for less demanding applications at higher temperatures [1-4]. Zirconium is a suitable alloying element in nickel alloys designed for high temperature applications. The zirconium content in $Ni_3Al$-based alloys may vary depending on the use of other alloying elements. The research has shown that alloys alloyed with zirconium have an increased intergranular strength and limited segregation of impurities at the grain boundaries. It is possible to eliminate the influence of sulphur, which in the subsequent reaction forms sulphides, which can cause high temperature cracking. Zirconium in intermetallic alloys also helps to increase ductility at high temperatures while increasing resistance to high temperature creep [5,6]. Other research results have shown that heat treatment is indispensable for process for production of the Ni-Al-Zr intermetallic alloys. Heat treatment of alloys has a principal influence on the microstructure and mechanical properties of the resulting product [6-8]. For the alloys containing 0.26 and 0.41 at.% Zr, it was determined that the structure contained γ-$Ni_5Zr$ eutectics [9]. Study the hot deformation behavior of Ni3Al-based alloy, hot compression tests were conducted in the temperature range of 1050 -1250 °C with the strain rates 0.01-10 $s^{-1}$. With the increase in deformation temperature and the decrease in strain rate, flow stress of the $Ni_3Al$-based alloy would be decreased [10].

## 2. EXPERIMENTAL

For experiment were used nickel alloys with different content of Zr and Al. The contents of these elements were 0.5 or 1.0 at.% of zirconium and 22 or 24 at.% of aluminum. The $Ni_3Al$ based alloys were vacuum induction melting in equipment Supercast 13. A part of castings were directionally solidified (DS) by Bridgman's method in tubes with specified apex angle (**Figure 1**). The samples were melting and solidified under a 5N argon atmosphere. The solidification rate was 50 mm/h. Directional solidification carried out with the use of the equipment Clasic CZ and Linn FRV-5-40/550/1900. Alloys were tested in as-cast (C) state and directed state (S). The prepared samples were used for determination of the selected mechanical properties in compression. Characterization of Ni-Al-Zr alloys is shown in **Table 1**.


**Figure 1** Sample of Ni-Al-Zr alloy in directed state

The micro-hardness values presented in **Table 1** show that the micro-hardness of these alloys is dependent on method of the sample preparation and on the zirconium content in the alloy. The directed samples appear to exhibit lower micro-hardness than the samples that have as-cast structure. The zirconium content in the investigated alloys has also an effect on micro-hardness, which is not so significant. The highest micro-hardness was achieved in the cast sample 2C with the highest aluminium and zirconium content. The lowest values had the directionally solidified samples containing 0.5 at.% Zr. Micro-hardness is therefore higher in the cast samples and also in the samples with higher zirconium contents. The indents after the measurement are shown in **Figure 2**.

**Table 1** Characterization of Ni-Al-Zr alloys

| Alloy | Sample No. | Composition (at.%) | Rate of DS (mm/h) | Microhardness HV0.05 |
|---|---|---|---|---|
| 1 | 1C | Ni-24Al-0.5Zr | - | 260 ± 6 |
| | 1S | | 50 | 239 ± 13 |
| 2 | 2C | Ni-24Al-1Zr | - | 271 ± 10 |
| | 2S | | 50 | 241 ± 13 |
| 3 | 3C | Ni-22Al-0.5Zr | - | 261 ± 10 |
| | 3S | | 50 | 235 ± 07 |
| 4 | 4C | Ni-22Al-1Zr | - | 265 ± 10 |
| | 4S | | 50 | 246 ± 12 |

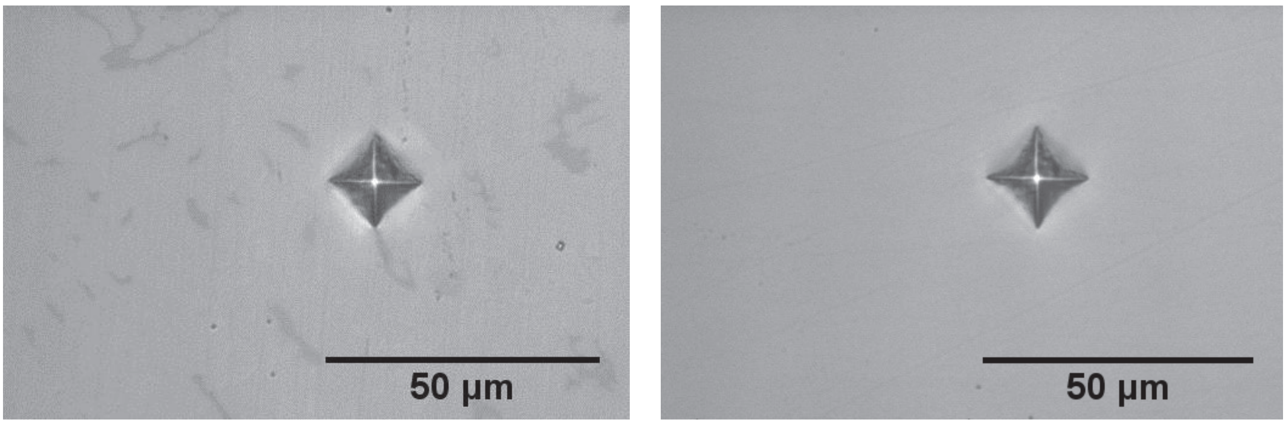


**Figure 2** Measurement of microhardness: Ni-24Al-1Zr alloy - as-cast (left) and directed state (right)

### 2.1. Mechanical properties in compression

The cylinders with a height of 12 mm and a diameter of 8 mm were used for testing of mechanical characteristics in compression. The plastometer GLEEBLE 3800 was used for isothermal tests by uniaxial compression. The test temperature was 800 °C with a heating rate of 3 °C/s. The heating to the test temperature was followed by a 15-second dwell. The chosen strain rate was $5 \cdot 10^{-2}$ $s^{-1}$. The tests were performed till the height deformation of 0.5. The yield strength $R_p$ was determined from the obtained values of the real stress. Moreover, the following indicative values were also determined $\sigma_{max}$ (maximum peak value of the real stress) and $\varepsilon_{max}$ (true strain corresponding to the $\sigma_{max}$). **Table 2** summarises the values of mechanical characteristics in compression tests at 800 °C. **Figure 3** shows compressive stress-strain curves.

**Table 2** Compressive mechanical characteristics

| Sample | C/S | $R_p$ (MPa) | $\sigma_{max}$ (MPa) | $\varepsilon_{max}$ (-) |
|---|---|---|---|---|
| 1C | C | 637 | 1274 | 0.33 |
| 1S | S | 780 | 1021 | 0.18 |
| 2C | C | 667 | 1283 | 0.35 |
| 2S | S | 880 | 1261 | 0.25 |
| 3C | C | 625 | 1216 | 0.34 |
| 3S | S | 640 | 1091 | 0.28 |
| 4C | C | 635 | 1129 | 0.32 |
| 4S | S | 745 | 1156 | 0.37 |

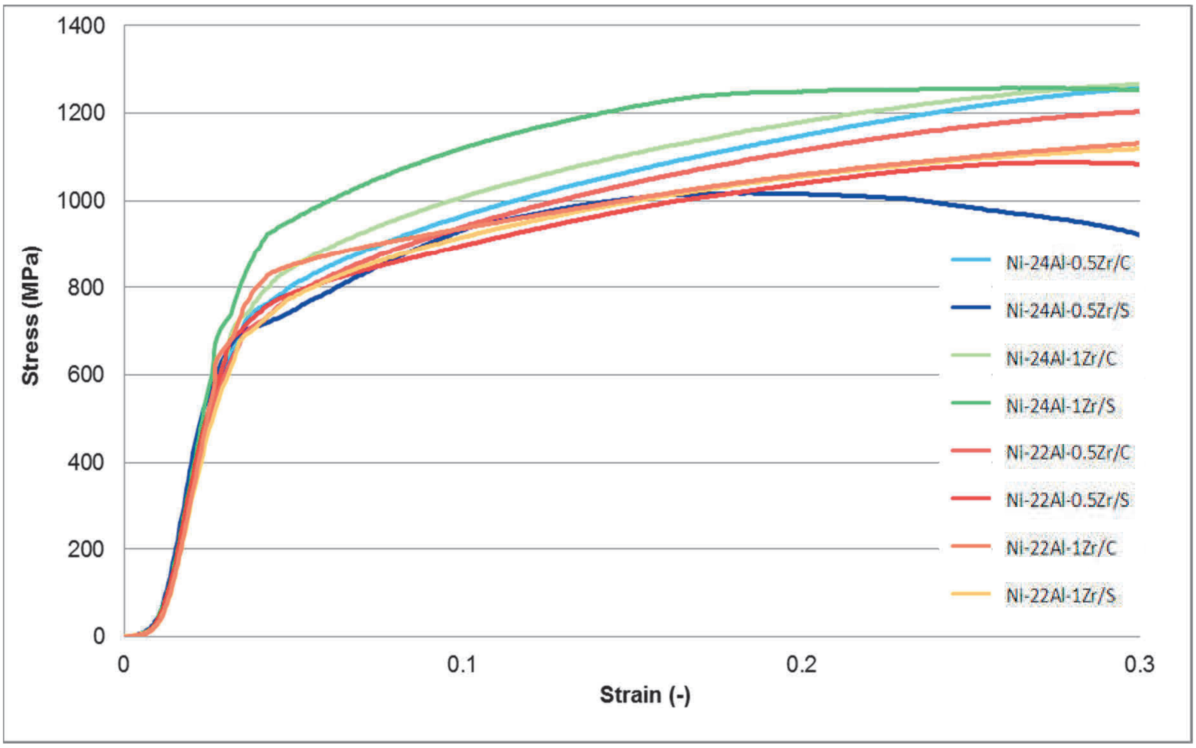


**Figure 3** Compressive stress-strain curves at 800 °C

It can be stated from the established values found, that the test samples in the as-cast state achieved higher values of maximum stress, but lower values of yield strength than the samples that were directionally solidified. In both types of Ni-Al-Zr samples mechanical properties increases with a simultaneous increase of percentage of zirconium and aluminum contents. The directionally crystallised alloy with the highest content of aluminum and zirconium reached an yield strength of approx. 800 MPa and the maximum actual stress value reached over 1200 MPa. On the other hand, the alloy with the same composition, but in the as-cast state, with the highest contents of aluminum and zirconium, reached a yield strength of approx. 600 MPa and a maximum stress of approx. 1300 MPa. Evolution of stress-strain curves under compression in as-cast and solidified samples has very similar character. It seems that solidified alloys have a higer yield strength. The solidified alloy Ni-24Al-1Zr exhibits the best values, namely a yield strength 880 MPa and maximum peak value of the real stress $\sigma_{max}$ 1261 MPa with a true strain corresponding to the $\sigma_{max}$ 0.25. Mechanical compression characteristics of similar alloys [11] were established already previously. Compared to the alloys based on $Ni_3Al$ and Ni-Al-Mo, the Ni-Al-Zr based alloys show better values of yield strength and of the maximum achieved stress.

## 2.2. Evaluation of structural characteristics

The structure of the as-cast alloys is dendritic. **Figure 4** shows macrostructures of alloys in the directed state in cross (circular views) and longitudinal sections (rectangular views). It is evident on the transversal and longitudinal cuts, that structure of solidified samples is formed by long columnar grains. **Figure 5** shows selected micro-structures of alloys containing 22 at.% of Al and zirconium. This alloy is formed by several phases.

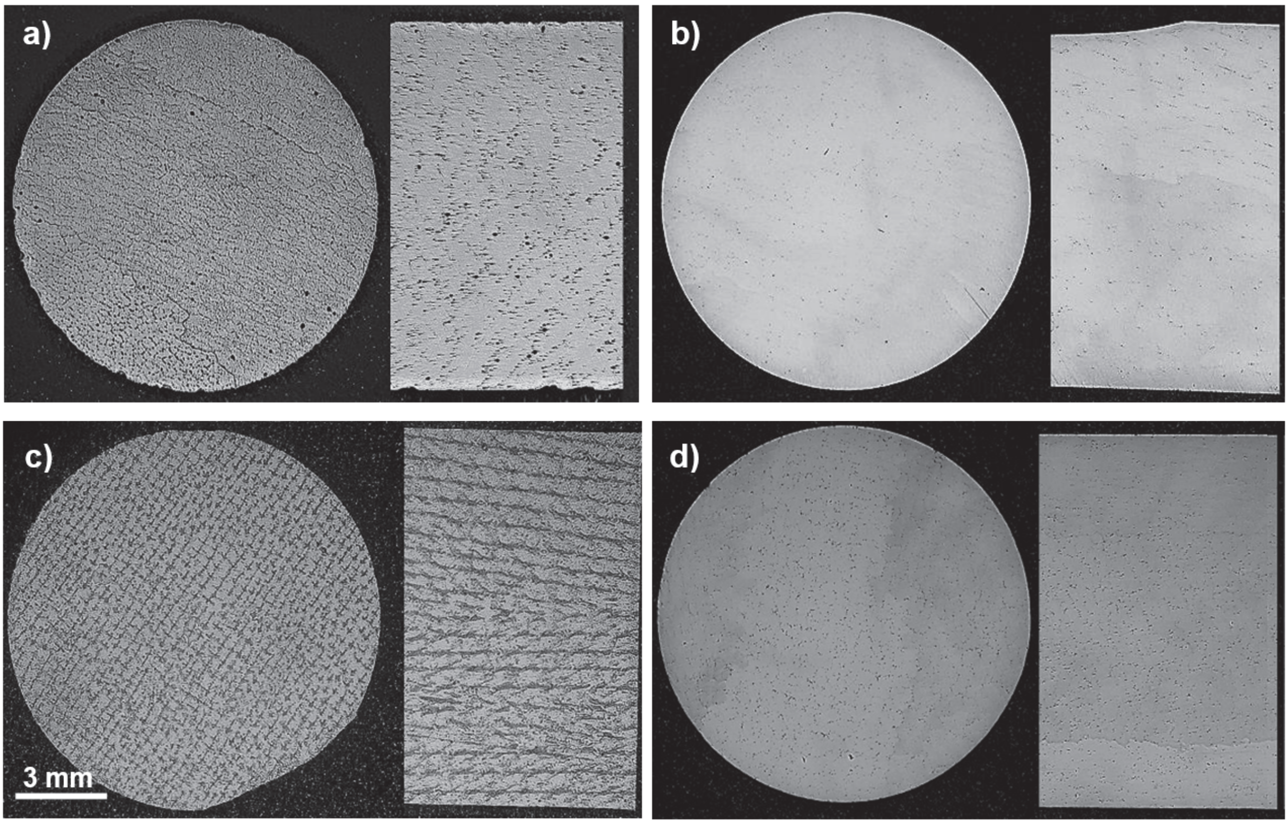


**Figure 4** Macrostructures of the samples in directed state, transversal and longitudinal direction: a) 1S, b) 2S, c) 3S, d) 4S

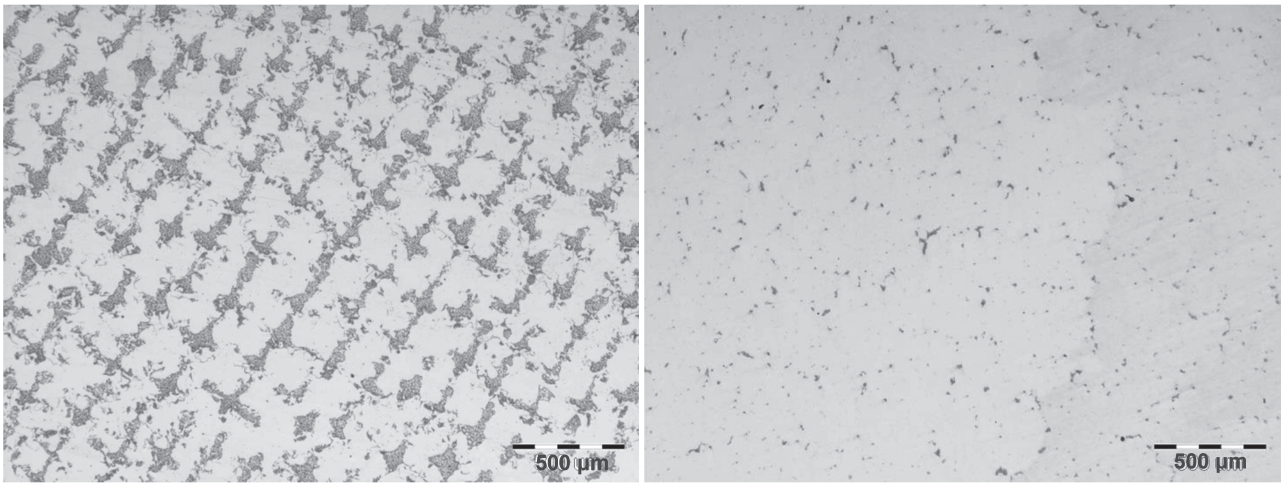


**Figure 5** Microstructures of the samples in directed state, transversal:
a) 3S, d) 4S

An EDS analysis was performed in order to verify the composition of the alloys and for the determination of the phases occurring in the structure. It has been found that the average chemical composition of the prepared experimental alloys does not differ fundamentally from the desired chemical composition. **Table 3** presents the average values of Ni, Al and Zr elements. Four types of phases were identified in the structures. The alloys Ni-Al-Zr are formed by $Ni_3Al$ and eutectic regions with Zr-rich phase. Phase $Ni_3Al$ occurs with different content of zirconium - without Zr and with dissolved zirconium (about 2-3 at.% of Zr). Zr-rich phase occurs in all type of the alloys with content of Zr about 13-17 at.%. It was established with the use of the ternary diagram [12], that it was probably the phase $Ni_5Zr$. The hypostoichiometric alloys are formed by (Ni) phase too. The set values of Ni and Al do not correspond completely to the assumed content for solid Ni solution according to the binary diagram [13]. With regard to previous TEM analyse [14,15], where individual phases including the network were determined very precisely, it can be assumed that this was the phase (Ni). This phase, however, has very small dimensions and it contains $Ni_3Al$ precipitates. For this reason, the measurement might have been influenced by the surrounding phase and by very small $Ni_3Al$ particles occurring within the mesh, thereby increasing the aluminium content. The sample 2C in the as-cast state contains sporadically the phase with composition Ni63.32-Al36.68 at.%. This phase according to the binary diagram corresponds to the $Ni_5Al_3$ phase, which can occur. The views and description of individual phase in the alloys Ni-24Al-0.5Zr, Ni-24Al-1Zr and Ni-22Al-0.5Zr are presented **in Figures 6 a-c.**

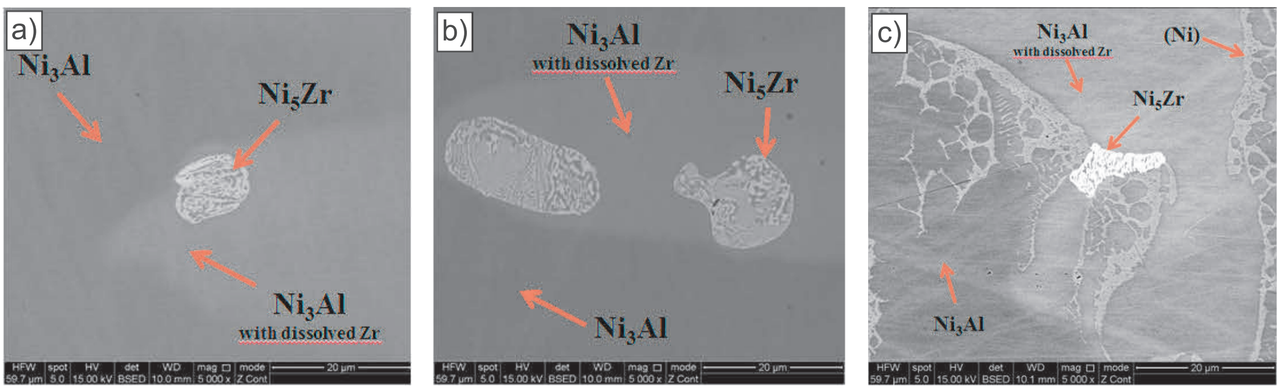


**Figure 6** View and description of phases of the alloys 1S, 2S and 3S

**Table 3** Chemical analysis of individual phases (EDS)

| Alloy | Average content of elements (at.%) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Ni | Al | Zr | Ni | Al | Zr | Ni | Al | Zr | Ni | Al | Zr |
| Ni-24Al-0.5Zr | 73.38 | 26.42 | 0.16 | 73.84 | 23.87 | 2.29 | 81.96 | 2.41 | 15.63 | - | - | - |
| Ni-24Al-1Zr | 73.71 | 26.02 | 0.27 | 73.75 | 23.63 | 2.62 | 79.46 | 3.44 | 17.10 | - | - | - |
| Ni-22Al-0.5Zr | 74.57 | 25.22 | 0.21 | 75.41 | 22.29 | 2.30 | 83.22 | 3.21 | 13.57 | 84.16 | 15.85 | - |
| Ni-22Al-1Zr | 74.97 | 24.69 | 0.34 | 75.48 | 22.61 | 1.91 | 84.03 | 1.54 | 14.43 | 83.38 | 16.12 | - |
| Phase | $Ni_3Al$ | | | Zr-rich $Ni_3Al$ | | | $Ni_5Zr$ | | | (Ni) | | |

## 2.3. Evaluation of structure after compression tests

**Figure 7** shows the samples after compression tests performed at a temperature of at 800 °C. **Figure 7** shows them in the directed state. It can be seen from the shape of the samples that the alloys have a fragile character of a fracture.

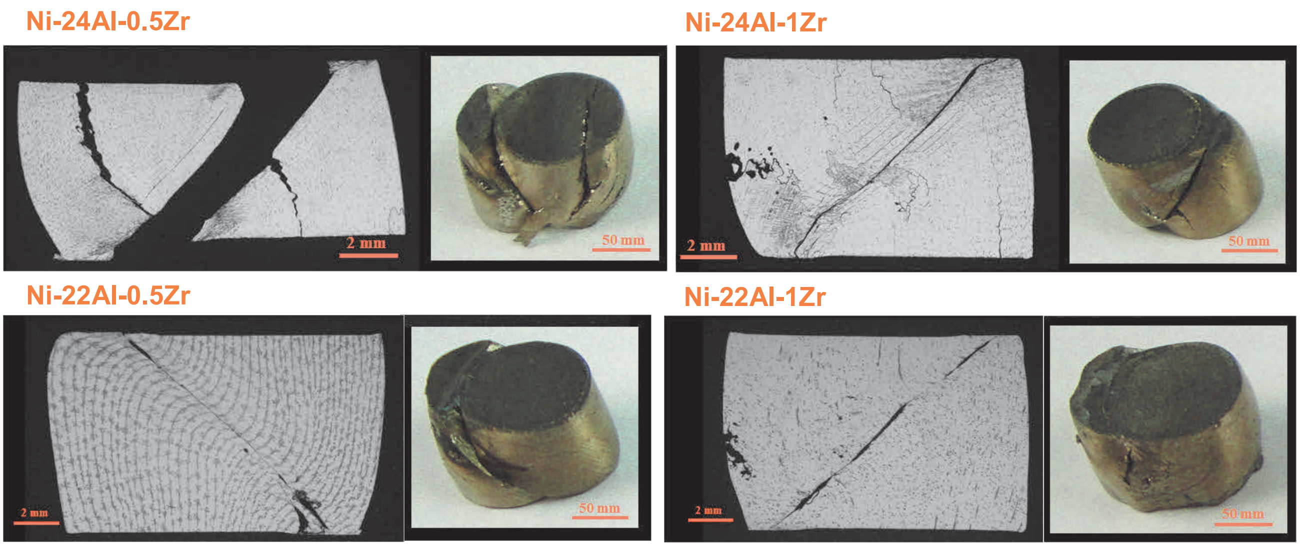


**Figure 7** The samples after compression tests - directed state

## 3. CONCLUSION

The alloys exhibit good values of yield strength and maximal achieved stress for this type of material. The alloys in directed state have better values of yield strength then as-cast alloys. The micro-hardness values show that the micro-hardness of these alloys is dependent on method of the sample preparation and on the zirconium content in the alloy. The samples in the as-cast state achieved higher values of maximum stress, but lower values of yield strength than the samples that were directionally solidified. In both types of Ni-Al-Zr samples mechanical properties increases with a simultaneous increase of percentage of zirconium and aluminum contents. The directed samples appear to exhibit lower micro-hardness than the samples that have as-cast structure. The structure of alloys depends on the aluminum content. The alloys with 24 at.% Al are formed by phases $Ni_3Al$ and Zr-rich phase $Ni_5Zr$. The hypostoichiometric alloys are formed by phases $Ni_3Al$, (Ni) and Zr-rich phase $Ni_5Zr$. Structure of solidified samples is formed by long columnar grains. The solidified alloys have a fragile character of a fracture.

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## REFERENCES

[1] WESSEL J. K. *Handbook of Advanced Materials: Enabling New Designs*. USA, A John Wiley & Sons, Inc., 2004. 645 p.

[2] JÓŹWIK, P., POLKOWSKI, W. and BOJAR, Z. Applications of $Ni_3Al$ based intermetallic alloys. Current stage and potential perceptivities. *Materials.* 2015. vol. 8, pp. 2537-2568.

[3] STOLOFF, N. S. and SIKKA, V. K. *Physical Metallurgy and Processing of Intermetallic Compounds*. Springer Science & Business Media. 2012. 684 p.

[4] SIKKA, V. K. et al. Advances in processing of $Ni_3Al$-based intermetallics and applications. *Intermetallics*. 2000. vol. 8, no. 9-11, pp. 1329-1337.

[5] JÓŹWIK, P. and BOJAR, Z. Influence of heat treatment on the structure and mechanical properties of $Ni_3Al$ - based alloys. *Archives of Metallurgy and Materials*. 2010. vol. 55, no. 1, pp. 271-279.

[6] LI, Yu-fang, GUO, Jian-ting and SHEN, Yi-fu. Influence of recrystallization and environment on tensile behavior of cold-rolled $Ni_3Al(Zr)$ alloys. *Transactions of Nonferrous Metals Society of China*. 2006. vol. 16, no. 2, pp. 368-375.

[7] HYJEK., P., SULIMA, I. and WIERZBIŃSKI, S. Ductilization of $Ni_3Al$ by alloying with boron and zirconium. *Archives of Materials Science and Engineering*. 2009. vol. 9, no. 2, pp. 69-74.

[8] PODANY, P., NOVY, Z. and DLOUHY, J. Recrystallization behaviour of a nickel-based superalloy. *Materiali in tehnologie*. 2016. vol. 50, no. 2, pp. 199-205.

[9] MOTEJADDED, H.B., SOLTANIEH, M. and RASTEGARI, S. Dissolution Mechanism of a Zr Rich Structure in a $Ni_3Al$ Base Alloy. *J. Mater. Sci. Technol.* 2011. vol. 10, no. 27, pp. 885-892.

[10] WU, I. et al. Deformation behavior and processing maps of $Ni_3Al$-based superalloy during isothermal hot compression. *Journal of Alloys and Compounds*. 2017. no. 712, pp. 687-695.

[11] MALCHARCZIKOVÁ, J. et al. Deformation behaviour of $Ni_3Al$ based alloys in compression. In *METAL 2016*: *25th International Conference on Metallurgy and Materials*. Ostrava: TANGER, 2016. pp. 1486-1491.

[12] *Ni-Al-Zr Phase Diagram, ASM Alloy Phase Diagrams Database* [online]. Materials Park, Ohio, USA: ASM International, 2009. [viewed 2009-01-04]. Available from https://www.asminternational.org/.

[13] MICHNA, Š. Aluminium materials and technologies from A to Z. Prešov: Adin. 2007. 613 p.

[14] MALCHARCZIKOVÁ, J. and KURSA, M. Non-alloyed $Ni_3Al$ based alloys - preparation and evaluation of mechanical properties. *Metalurgija*. 2013. vol. 52, no. 3, pp. 309-312.

[15] MALCHARCZIKOVÁ, J. and KURSA, M. Structural characteristics of $Ni_3Al$ based alloys depending on the preparation conditions. *Metalurgija*. 2015. vol. 54, no. 4, pp. 635-638.