A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 55 2010 Issue 1

B. ZYŚK*, M. KULCZYK**, M. LEWANDOWSKA*, K. J. KURZYDŁOWSKI*

EFFECT OF HEAT TREATMENT AND HYDROSTATIC EXTRUSION ON MECHANICAL PROPERTIES OF A CuCrZr ALLOY

WPŁYW OBRÓBKI CIEPLNEJ I WYCISKANIA HYDROSTATYCZNEGO NA WŁAŚCIWOŚCI MECHANICZNE STOPU CuCrZr

In this work the effects of heat treatment and hydrostatic extrusion on mechanical properties of a CuCrZr alloy were investigated. The samples of CuCrZr alloy were subjected to solution annealing at α temperature of 1000°C for 1h followed by water quenching. This was followed by aging at temperatures of 350, 450, 480, 550 and 650° C for the time raging from $^1/_2$, to 3 hours. Two samples: (1) solution annealed and water quenched and (2) solution annealed, water quenched and aged at 480°C for 2 hours were subjected to the process of hydrostatic extrusion. The results obtained show that ageing increases the yield strength of the alloy by 500% whereas plastic deformation by hydrostatic extrusion by 1000%. The synergic effects of precipitation and deformation strengthening were also observed on samples subjected to ageing prior to or after hydrostatic extrusion. However, it is suggested that the two strengthening mechanisms are not fully additive.
Keywords: CuCrZr alloys, thermal treatment, hydrostatic extrusion

W pracy badano wpływ obróbki cieplnej i wyciskania hydrostatycznego na właściwości mechaniczne stopu CuCrZr. Próbki ze stopu CuCrZr poddano przesycaniu z temperatury 1000°C w wodzie. Następnie były one starzone w temperaturze 350, 450, 480, 550 i 650°C w czasie od $^1/_2$ do 3 godzin. Dwie próbki: (1) przesycana i (2) starzona w temperaturze 480°C przez 2 godziny zostały poddane procesowi wyciskania hydrostatycznego. Uzyskane wyniki wskazują, że starzenie powoduje wzrost plastyczności stopu o 500%, podczas gdy po odkształceniu plastycznym metodą wyciskania hydrostatycznego wzrasta o 1000%. Synergiczny efekt umocnienia wydzieleniowego i odkształceniowego zaobserwowano w próbkach poddanych starzeniu zarówno przed jak i po wyciskaniu hydrostatycznym. Uzyskane wyniki wskazują jednak, że obydwa mechanizmy umocnienia nie są w pełni addytywne.

1. Introduction

CuCrZr alloys combine high mechanical strength and good thermal conductivity which make them attractive candidates for such field of application as heat removal elements. Good mechanical properties of these alloys are obtained due to precipitation strengthening via heat treatment, which consists of solution annealing followed by water quenching and aging [1,2]. Progress made in nano-refinement of metals calls for investigations whether or not these properties can be further improved by grain size strengthening.

Various techniques, which can be used to refine the size of grains in polycrystalline material to below 100 nm, have been proposed recently. A large group of these methods is based on Severe Plastic Deformation, SPD, which enables micro-grained aggregates to be transformed to the nano-size by the accumulation and rearrangement/annihilation of the crystal lattice defects, primarily dislocations. Experimental observations showed, that for such a transformation to take place, a large degree of plastic deformation is required, which usually exceeds the maximum equivalent strain achievable in simple plastic forming methods [3,4]. As a result, special deformation methods have been employed, including Equal Channel Angular Pressing [5], High Pressure Torsion [6], Cyclic Extrusion Compression [7] and Multi Axes Forging [8]. More recently, Hydrostatic Extrusion, HE, has been used for this purpose [9,10].

The efficiency of HE in the process of grain refinement has been already confirmed for a number of materials, including aluminium alloys [10,11], titanium [12] and steels [13,14]. However, copper and its alloys were studied much less extensively. The aim of this work is to apply hydrostatic extrusion to reduce the grain size in CuCrZr alloy and to explore possibility for combined grain boundary and precipitation strengthening.

* FACULTY OF MATERIALS SCIENCE AND ENGINEERING, WARSAW UNIVERSITY OF TECHNOLOGY, 02-507 WARSZAWA, 141 WOLOSKA STR., POLAND
** POLISH ACADEMY OF SCIENCES. INSTITUTE OF HIGH PRESSURE PHYSICS (UNIPRESS), 01-142 WARSZAWA, 29/37 SOKOŁOAWSKA STR., POLAND

2. Material and experimental

A CuCrZr alloy (trade name MHY) with the chemical composition given in Table 1 was used, delivered in the form of extruded rods with a diameter of 20 mm.

TABLE 1

The chemical composition of the CuCrZr alloy

chemical composition	Cr	Zr	Sb	Zn	Fe	P	Cu	others
according to a standard	0.4 ÷ 1.0	0.02 ÷ 0.15	max 0.01	max 0.02	max 0.05	max 0.01	balance e	max 0.2
measured value	0.7	0.08	0.01	0.01	0.03	0.01	balance e	0.09

In order to determine the effect of heat treatment on mechanical properties of the CuCrZr alloy, the samples were subjected to solution annealing at temperature 1000°C for 1h followed by water quenching. They were subsequently aged at temperatures of 350, 450, 480, 550 and 650°C for $^1/_2$, 1, 2 and 3 hours. After ageing the samples were cooled in air.

Billets of the CuCrZr alloy were also subjected to HE processing. This process was carried out in three consecutive passes, starting from a diameter of 20 mm, down to 10, 5 and 3 mm. These correspond to a true strain of 1.4. 2.8 and 3.8, respectively.

Mechanical properties of CuCrZr alloy in the as delivered state, after heat treatment and HE processing were evaluated by hardness measurements and in tensile tests. Microstructure was studied using light and electron microscopy (transmission. TEM and scanning. SEM) equipped with EDX detector for local chemical analysis. The microstructures revealed by microscopic observations were described quantitatively in terms of the grain size and shape. To describe the grain size, the equivalent diameter of grains, d, defined as the diameter of a circle which has the surface area equal to the surface area of a given grain, was measured. The average equivalent diameter E(d) and variation coefficient CV(d) defined as the ratio of the standard deviation to the mean value, were determined. The grain shape was described by shape parameter defined as the ratio of the maximum diameter to the equivalent diameter of a given grain, d_{max}/d, which quantifies the elongation of a grain.

3. Results

3.1. Effect of heat treatment on mechanical properties of CuCrZr alloy

The results of hardness measurements as a function of temperature and ageing time are given in Table 2. It has been found that the samples subjected to solution annealing and water quenching exhibit very low hardness of 42 HV30. Ageing at a temperature of 350°C does not cause a significant change in the hardness, which means that this temperature is too low to induce the precipitation of second phase particles. The highest strength was obtained in the case of ageing at a temperature of 480°C for 2h. It has been also observed that ageing at temperatures higher than 500°C results in a decrease in strength.

TABLE 2

Results of hardness measurements as a function of temperature and aging time

time [h]	0	0.5	1	2	3
ageing 350°C	42	48	44	45	45
ageing 450°C	42	93	106	107	89
ageing 480°C	42	105	110	118	117
ageing 550°C	42	85	93	84	86
ageing 50°C	42	68	73	66	67

For further examinations, two samples have been selected: (1) solution annealed and water quenched and (2) solution annealed, water quenched and aged at 480°C for 2 hours. Microstructure of a sample after quenching consists of equiaxed grains (Fig. 1a) with an average equivalent diameter of 50 μm. TEM observations revealed the presence of relatively large particles – 0.5 μm in diameter (Fig. 1b) located at grain boundaries and in grain interiors. Analysis of their chemical composition indicates that they are primary particles of chromium. Ageing does not cause any significant changes in the grain size, which remains at the level of 50 μm (Fig. 2a). However. ageing at 480°C for 2 hours results in the formation of small (about ten nanometers in size) GP zones evenly spaced in the matrix (Fig. 2b).

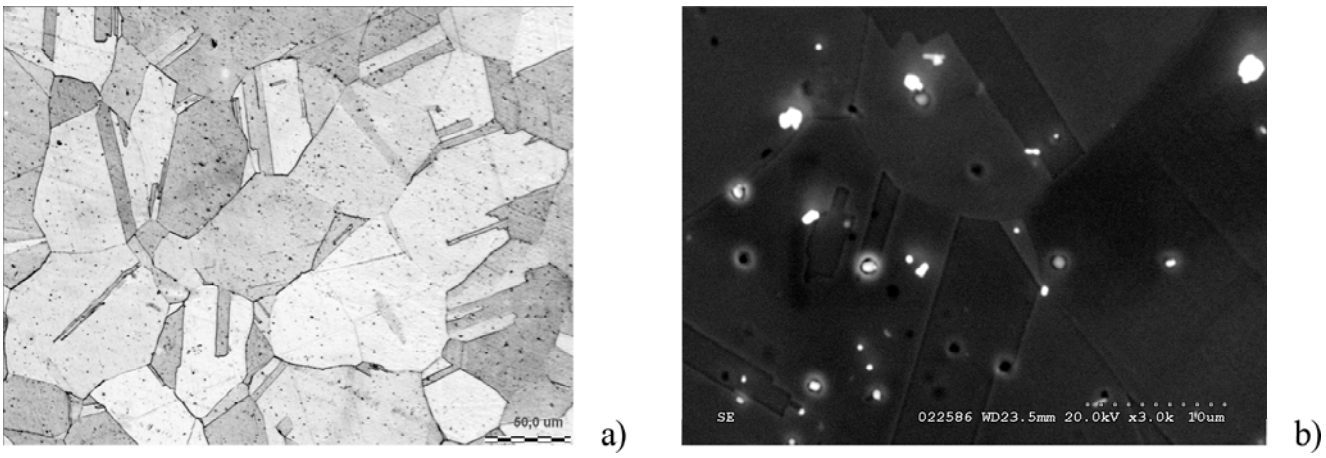

Fig. 1. Microstructure of the as-quenched sample: light microscopy (a), scanning electron microscopy (b)

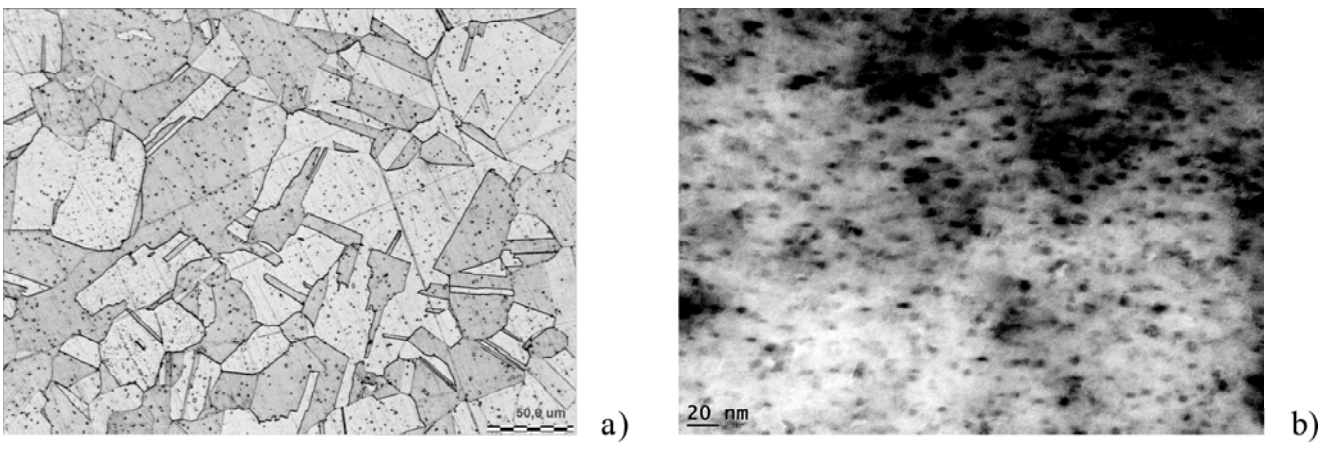

Fig. 2. Microstructure of the sample aged at 480°C for 2 hours: light microscopy (a), transmission electron microscopy (b)

3.2. Effect of hydrostatic extrusion on microstructure and mechanical properties of the CuCrZr alloy

Two billets were subjected to the process of hydrostatic extrusion. TEM images of microstructure after hydrostatic extrusion are shown in Fig. 3. The microstructure of HE CuCrZr is built up with dislocation cells, on average 200 nm in size in the case of samples extruded in water quenched state and 170 nm for aged one. The microstructure images were analyzed quantitatively, to determine the average grain size E(d), grain size variation coefficient CV(d) and grain shape factor. The measured values of these parameters are summarized in Table 3. It can be noted that hydrostatic extrusion results in a significant structure refinement (Fig. 3).

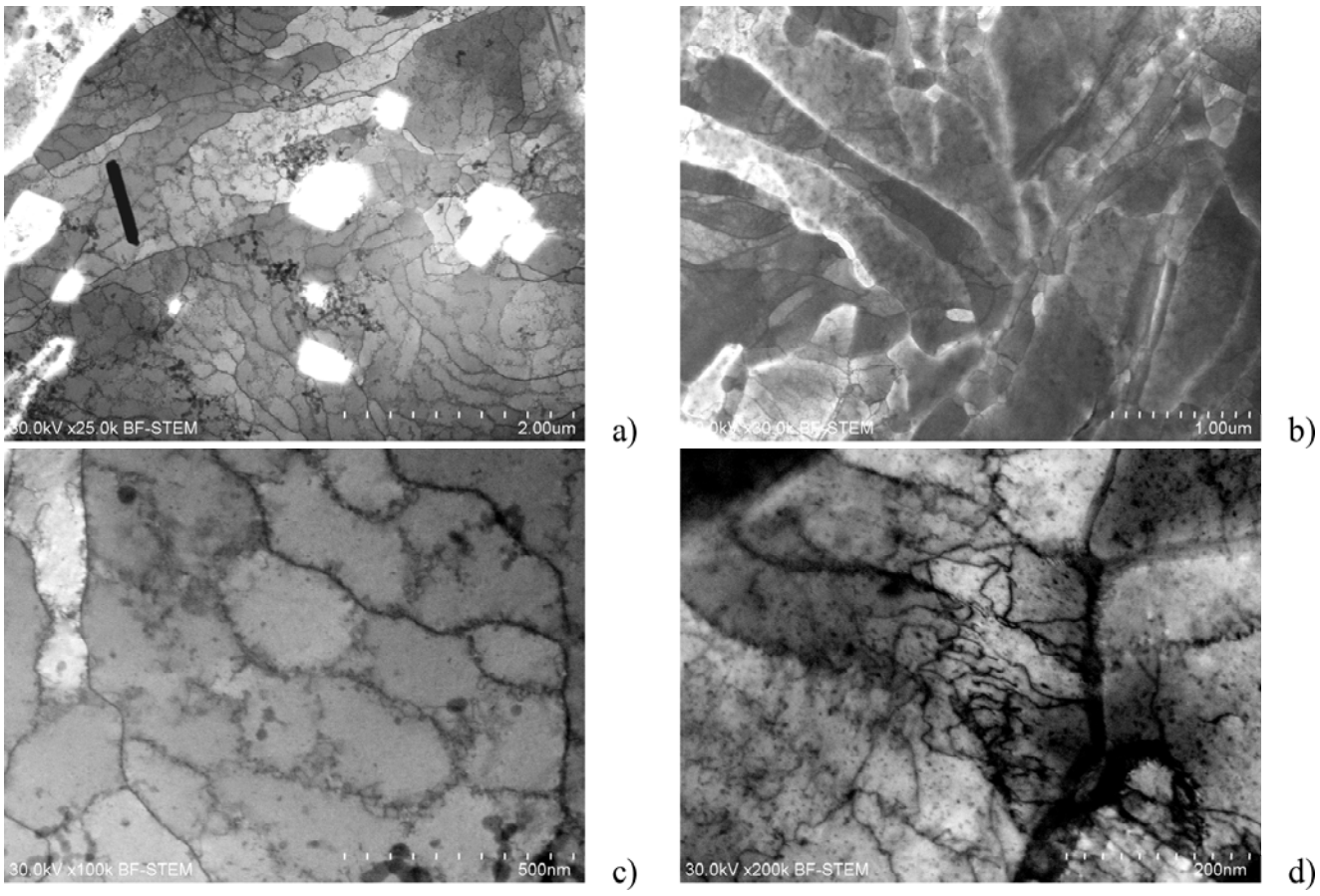

Fig. 3. Microstructures of samples subjected to hydrostatic extrusion: (a, c) after quenching and (b, d) ageing at temperature 480°C for two hours

TABLE 3

The average grain size for samples subjected to hydrostatic extrusion

sample state	parameter	before hydrostatic extrusion	after hydrostatic extrusion
solution annealing 1000°C/1h	E (d) [μm]	51.47	0.20
	CV (d)	0.41	0.50
	α	1.27	1.65
ageing 480°C/2h	E (d) [μm]	52.61	0.17
	CV (d_2)	0.47	0.59
	α	1.31	1.65

As HE processing proceeds at a very high strain rate, adiabatic heating phenomena are likely to take place. On the other hand, estimated temperature rise during individual extrusion passes are much below the aging temperature. This suggests that no change in precipitates should occur during HE. This is supported by microstructure observations (Fig. 3 c, d) which give no evidence of precipitate in the sample extruded immediately after quenching. No significant changes in precipitates size can be observed in the sample extruded after ageing.

On the other hand, HE alters large primary Cr precipitates. Size measurements of these particles revealed slight reduction in their size due to HE processing. The reduction in size is more pronounced in the case of sample extruded after ageing – see data in Table 4. It should also be noted that HE does not substantially affect spa-

tial distribution of these particles (see Fig. 4) which are fairly uniform before and after HE.

TABLE 4

The effect of hydrostatic extrusion on the size of primary Cr particles

sample	water quenched before HE	water quenched after HE	aged before HE	aged after HE
average particie size [μm]	0.57	0.54	0.54	0.44

TABLE 5

Mechanical properties of the CuCrZr alloy before and after hydrostatic extrusion

sample	UTS [MPa]	YS [MPa]	A_r [%]	A_c [%]
water quenched	226	54	27.2	38.6
water quenched and aged at 480°C for 2 hours	388	277	12.3	23.5
water quenched and hydrostatically extruded	527	510	0.4	8.2
water quenched. aged at 480°C for 2 hours and hydrostatically extruded	691	611	0.7	8.5

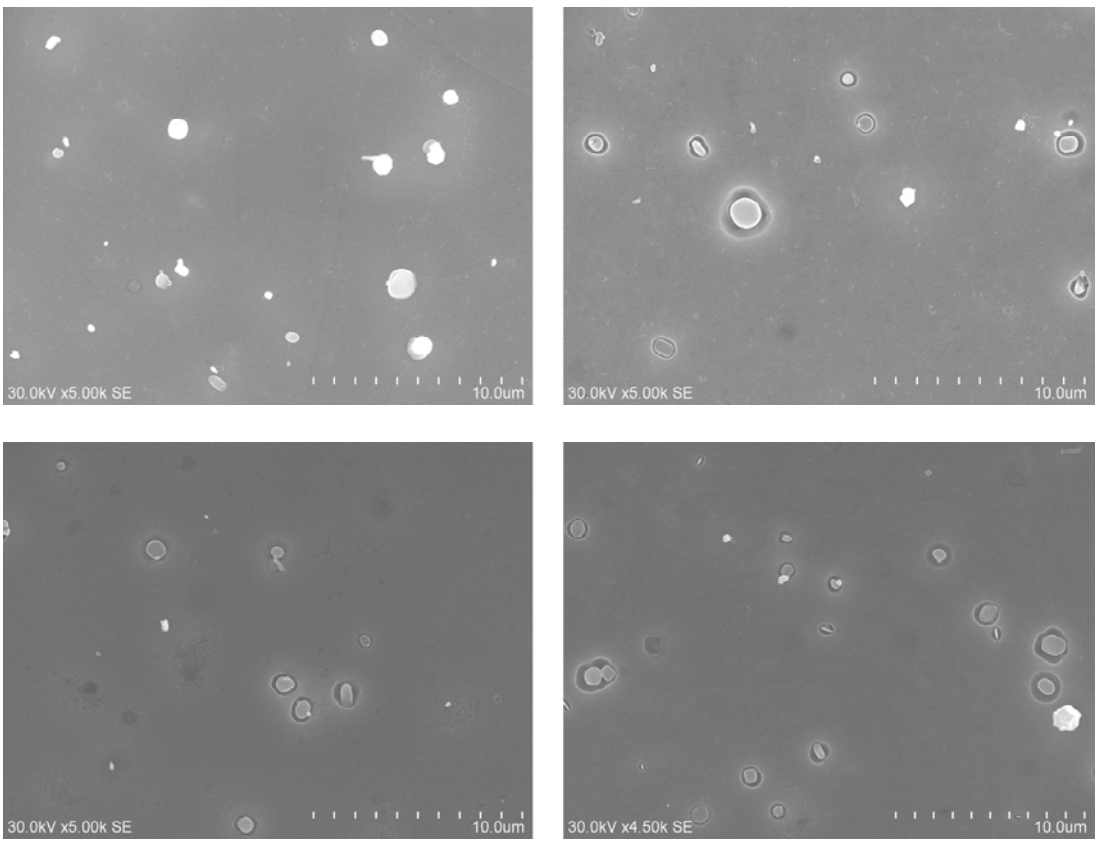

Fig. 4. Spatial arrangement of primary particles before and after hydrostatic extrusion

The results of tensile tests are summarized in Table 5, which lists values of such parameters as yield strength (YS), ultimate tensile strength (UTS), total elongation (A_c), and uniform elongation (A_r).

The sample immediately after quenching exhibits relatively low strength (the yield strength is only 54 MPa). It is worth noting that ageing at 480°C for 2 hours brings about a fivefold increase in yield strength (to 277 MPa). On the other hand, processing by hydrostatic extrusion increases it ten times. This shows that HE processing is much more efficient in hardening of CuCrZr alloy. The best combination of strength parameters was obtained for the sample which was hydrostatically extruded after ageing. In this sample, synergic effects of HE and ageing can be observed. However, these two effects are not additive. The synergy of these two strengthening mechanisms will be a subject of further investigations.

It should also be noted that significant improvement in mechanical strength is at the expense of ductility. The samples after HE exhibit particularly low uniform elongation (below 1 %). On the other hand, the total elongation to failure is much higher (about 8% for HE processed samples) which indicates reasonable plasticity with a strong tendency to strain localisation.

3.3. Ageing characteristics of hydrostatically extruded samples

The other possibility to combine strengthening effects arising from precipitates and HE is to age the samples after extrusion. Fig. 5 shows the changes in microhardness as a function of ageing time for two samples: (1) previously subjected to HE and (2) solution annealed and water quenched. For comparison, microhardness of the sample aged prior to hydrostatic extrusion is also presented. Ageing of the HE processed sample results in a significant increase in microhardness. It should be, however, noted that the maximum value of HV0.2 is obtained after 1 hour ageing. For longer ageing times, the microhardness decreases. This ageing characteristic is quite different from one for un-deformed sample, for which the maximum value is achieved after 2 hours.

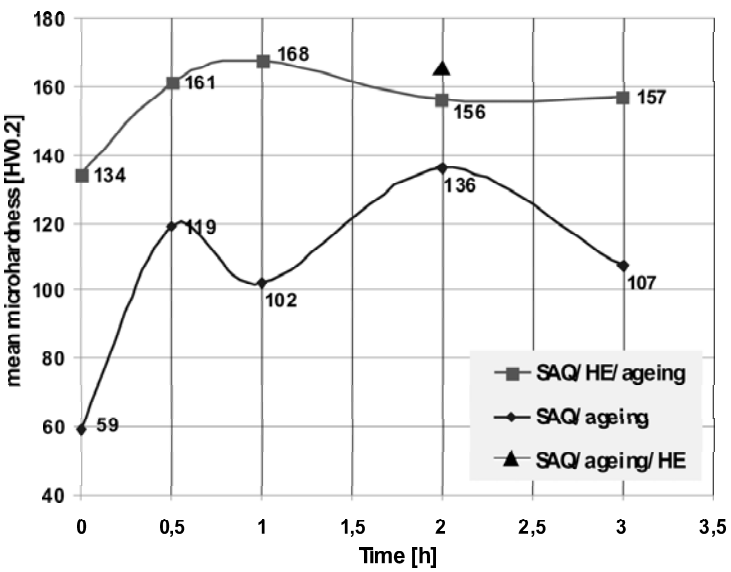

Fig. 5. Changes in microhardness as a function of ageing time (SAQ - solution annealing and water quenching)

4. Summary

In this work the effects of heat treatment and hydrostatic extrusion on mechanical properties of a CuCrZr alloy were investigated. It was revealed that ageing may increase the yield strength of the alloy by 500% whereas plastic deformation by hydrostatic extrusion by 1000%. The synergic effects of precipitation and deformation strengthening were also obtained in two ways, by ageing prior to and after hydrostatic extrusion. In both cases, the improvement of strength was observed. However, it is suggested that these two mechanisms are not fully additive.

Acknowledgements

This work was supported by Polish Ministry of Science and Higher Education (Grant No PBZ-MNISW-3/3/2006). HE experi-

ments were carried out at the Institute of High Pressure Physics of Polish Academy of Sciences within the project coordinated by the Faculty of Materials Science and Engineering of Warsaw University of Technology.

REFERENCES

[1] M. Merola, A. Orsini, E. Visca, S. Liberta, L. F. Moreschi, S. Storai, B. Panella, E. Campagnoli, G. Ruscica, C. Bosco, Influence of the manufacturing heat cycles on the CuCrZr properties, Journal of Nuclear Materials **307**, 311, 677-680 (2007).

[2] G. M. Kalinin, A. D. Ivanov, A. N. Obushev, B. S. Rodchenkov, M. E. Rodin, Y. S. Strebkov, Ageing effect on the properties of CuCrZr alloy used for the ITER HHF components, Journal of Nuclear Materials **367**, 370 920-924 (2007).

[3] R. Z. Valiev, R. K. Islamgaliev, I. V. Alexandrov, Bulk nanostructured materials from severe plastic deformation, Progress in Materials Science **45**, 103-189 (2000).

[4] D. A. Hughes, N. Hansen, Microstructure and strength of nickel at large strains, Acta Matetrialia **48**, 2985-3004 (2000).

[5] R. Z. Valiev, T. G. Langdon, Principles of equal channel angular pressing as a processing tool for grain refinement, Progress in Materials Science **51**, 881-981 (2006).

[6] G. Sakai, Z. Horita, T. G. Langdon, Grain refinement and super plasticity in an aluminum alloy processed by high-pressure torsion ,Material Science & Engineering **A 393**, 344-351 (2005).

[7] M. Richert, Q. Liu, N. Hansen, Microstructural evolution over a large strain range in aluminium deformed by cyclic-extrusion-compression, Material Science & Engineering **A 260**, 275-283 (1999).

[8] B. Cherukuri, T. S. Nedkova, R. Srinivasan, A comparison of the properties of SPD-processed AA 6061 by equal-channel angular pressing, multi axial compressions/forgings and accumulative roll bonding, Material Science & Engineering **A 410-411**, 394-397 (2005).

[9] M. Lewandowska, K. J. Kurzydłowski, Recent development in grain refinement by hydrostatic extrusion, Journal of Material Science **43**, 7299-7306 (2008).

[10] M. Lewandowska, W. Pachla, K. J. Kurzydłowski, Fabrication of high strength nanostructured aluminium alloys by hydrostatic extrusion, International Journal of Materials Research (formely Z.Metallkd), **98**, 172 (2007).

[11] M. Lewandowska, Microstructure evolution in age-hardenable aluminium alloy during processing by hydrostatic extrusion, Journal of Microscopy **224**, 34 (2006).

[12] W. Pachla, M. Kulczyk, M. Suś-Ryszkowska, A. Mazur, K. J. Kurzydłowski, Nanocrystalline titanium produced by hydrostatic extrusion, Journal of Materials Processing Technology **205**, 173-182 (2008).

[13] A. T. Krawczyńska, M. Lewandowska, K. J. Kurzydłowski, Nanostructure formation in austenitic stainless steel, Solid State Phenomena **140**, 173-178 (2008).

[14] M. Lewandowska, A. Krawczyńska, M. Kulczyk, K. J. Kurzydłowski, Structure and properties of nano sized Eurofer 97 steel obtained by hydrostatic extrusion, Journal of Nuclear Materials **386-388**, 499-502 (2009).

Received: 10 September 2009.