

# Influence of cyclic loading on the onset of failure in a Zr-based bulk metallic glass

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Received: 8 April 2014 / Accepted: 25 April 2014 / Published online: 26 June 2014  
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**Abstract** Samples of  $Zr_{62.5}Cu_{22.5}Fe_5Al_{10}$  bulk metallic glass are subjected to uniaxial compression. Comparison of tests in monotonic loading and cyclic loading (repeated loading to the onset of plasticity, then unloading) shows that the compressive plasticity of the glass is drastically reduced under cyclic loading. It is argued that this effect arises from stress reversal accelerating the concentration of shear on a dominant shear band. The link with compression–compression fatigue results, and the consequences for low-cycle fatigue of metallic glasses are considered.

## Introduction

Bulk metallic glasses (BMGs) are glassy (amorphous) alloys that can be cast with minimum dimension in the range 1 mm to 1 cm [1–3]. Because samples of significant size can be made, there has been much interest in exploring the use of BMGs as structural materials [4]. Compared to conventional polycrystalline alloys, BMGs exhibit high strength, hardness, wear resistance, and elastic strain limit [4, 5]. When heated into the supercooled liquid state near the glass-transition temperature  $T_g$ , they show good plastic

formability (fluidity) [6] which can be exploited both for such macroscopic processes as blow-molding [7] as well as precision forming on micrometer and even nanometer scales [8]. The combination of high strength and high formability is considered particularly attractive.

The plastic deformation of metallic glasses, including BMGs, is inhomogeneous when it occurs at relatively low homologous temperature (i.e., normalized with respect to  $T_g$ ) including room temperature [9, 10]. The inhomogeneous flow, localized in shear bands just 10–20 nm thick [11], is an unattractive feature as it is associated with strain-softening and essentially zero ductility. Although there is local plasticity in the shear bands, samples in tension fail catastrophically by shear along a dominant shear band. BMGs were originally considered to have relatively poor fatigue resistance, possibly also connected to the lack of any strain-hardening, but as pointed out in recent reviews, they in fact show a wide range of fatigue behavior influenced by intrinsic and extrinsic factors [12, 13]. There has been comparatively little work on compression–compression fatigue of BMGs. For a given stress amplitude, fatigue lives are expected to be longest in this case, since fatigue cracks remain closed under compressive load. Freels et al. [14] studied compressive–compressive fatigue of a Cu–Zr-based BMG, and found that fracture occurs in shear at roughly the same angle as seen in continuous monotonic compression. In that case, this is at 41° to the loading axis (i.e., the normal to the fracture surface is at 49° to the loading axis). Furthermore, they found that in fatigue, fragments are lost from the sample surface (“chipping”); the chips are bounded by shear planes at the same angle.

As yet there have been very few studies [15, 16] of low-cycle fatigue (LCF) of metallic glasses and BMGs. In this study, we conduct compression tests on a BMG,

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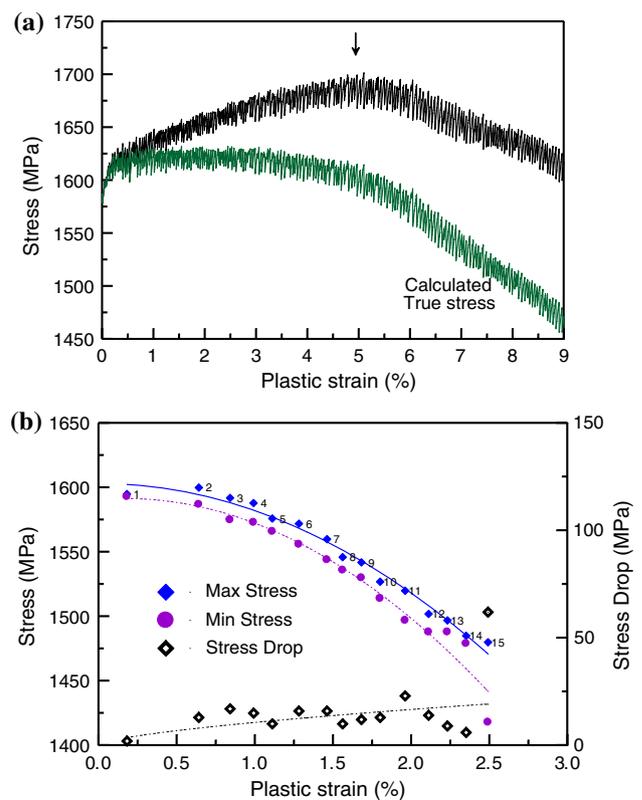
$Zr_{62.5}Cu_{22.5}Fe_5Al_{10}$  (composition in at. %), that shows exceptionally good plasticity (several percents) in compression [17]. Comparison of tests in monotonic loading and cyclic loading (repeated loading to the onset of plasticity, then unloading) shows that the compressive plasticity of the BMG is drastically reduced by the cyclic loading. The origins of this effect are explored. It is related to the reports of compression–compression fatigue, and the consequences for LCF resistance of metallic glasses are considered.

## Experimental methods

Rod-shaped  $Zr_{62.5}Cu_{22.5}Fe_5Al_{10}$  BMG samples 2 mm in diameter were prepared by arc-melting high-purity (99.9 %) elements in a Ti-gettered Ar protective atmosphere and subsequent injection casting into a copper mold. X-ray diffraction (XRD) with Cu  $K_\alpha$  radiation suggested that the as-cast samples are fully glassy, in agreement with earlier findings on this composition [17]. The rods were cut into lengths of 2, 3, or 4 mm to give cylindrical test samples (both ends polished to be parallel) of aspect ratio (height:diameter) of 1:1, 1.5:1, or 2:1. BN particles were sprayed on both sides between the sample and WC plates before the compressive tests as a lubricant to reduce the friction force. These were subjected to quasi-static (strain rate,  $5 \times 10^{-4} \text{ s}^{-1}$ ) compression at room temperature using a Shimadzu AG-X mechanical testing machine, for which the stiffness (frame rigidity) is  $42 \text{ kN mm}^{-1}$ . Both monotonic loading to failure and cyclic loading were used. In the latter case, loading in each cycle was up to the onset of plasticity indicated by the appearance of serrations in the stress–strain curve (i.e., stress drops). After the onset of plasticity, the sample was unloaded, either completely or partially, as described by the stress ratio  $r = \sigma_0/\sigma_{\max}$ . Mostly  $r = 0$  (i.e., complete unloading), but in some cases the applied stress was reduced not to zero but to 200 MPa, giving  $r \approx 0.12$ . Unloading was at a strain rate of  $\sim 10^{-3} \text{ s}^{-1}$ . The period before the start of the next loading cycle was  $\sim 100 \text{ s}$ . The cyclic loading frequency was  $\sim 3 \times 10^{-4} \text{ Hz}$ . The development of shear bands on the cylindrical surfaces of the samples, and the fracture surfaces after failure, were observed using scanning electron microscopy (SEM).

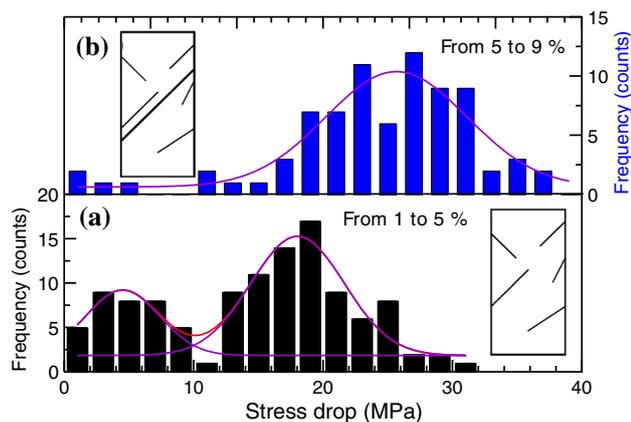
## Results

Figure 1a shows the compressive plasticity of a  $Zr_{62.5}Cu_{22.5}Fe_5Al_{10}$  BMG sample of 2:1 aspect ratio under the continuous test. Only the plastic strain region is shown. On monotonic loading, the engineering stress rises, reaches a maximum at a critical strain (arrowed) and then decreases.



**Fig. 1** Compression testing of  $Zr_{62.5}Cu_{22.5}Fe_5Al_{10}$  BMG. The samples are cylinders 2 mm in diameter and 4 mm in height (i.e., aspect ratio 2:1). **a** The stress–strain curves (nominal and calculated true stress) for monotonic loading to failure. The arrow indicates the strain at which there appears to be a change in deformation mode (stage 1 to stage 2). **b** The results of cyclic loading (the stress ratio  $r = 0$ ) are shown by the data points labeled with the cycle number. For each cycle, the maximum stress at the onset of yielding, the minimum stress at the end of the stress drop under load, and the value of the stress drop are given. All the results are shown as a function of plastic strain (i.e., total strain – elastic strain)

The true stress (calculated assuming macroscopically homogeneous deformation and constant sample volume during plastic deformation) stays approximately constant until the critical strain, beyond which it decreases. The tests were conducted on three samples, and all show similar two-stage behavior, though the critical plastic strain beyond which the second stage is reached is highly variable (from 1.25 to 6 %), as is the plastic strain to failure (up to 13 %). The monotonic loading stress–strain curves show evident serrations, i.e., stress drops. Figure 2 shows the distribution of magnitude of the stress drops in stages 1 and 2. In studies using video photography as the compression tests are conducted [18], stage 1 deformation is seen to occur through the progressive appearance of more shear bands that are relatively small and do not span the sample cross section (inset a). In stage 2, deformation takes place mostly through repeated stick–slip on one dominant shear band [19, 20] (or a pattern of very few bands, though new shear



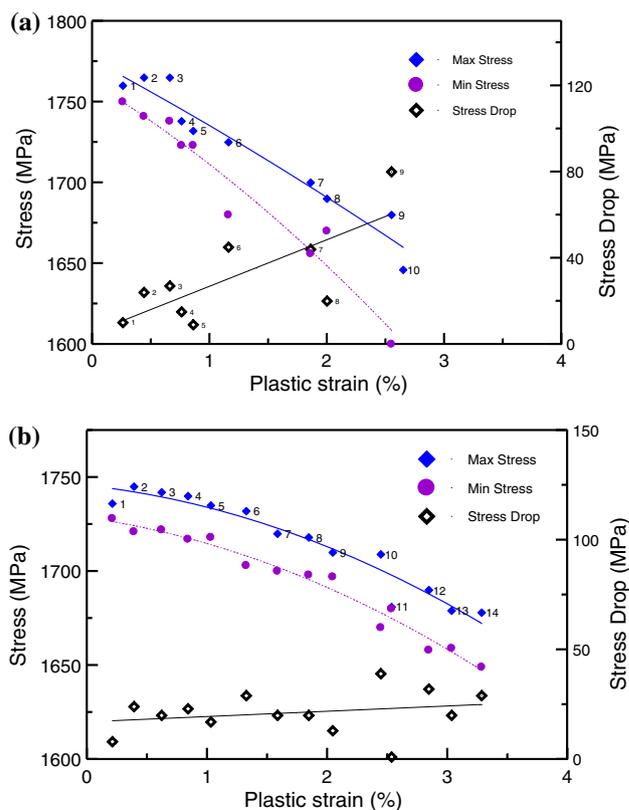
**Fig. 2** Deformation stages in the monotonic loading test in Fig. 1. **a** In stage 1 (plastic strain less than the critical, arrowed value of 5 %), the values of stress drop are bimodally distributed around 5.7 and 18.0 MPa. In this early stage of deformation, plastic flow is associated with the development of new shear bands that are rather uniformly distributed (see corresponding *inset*, showing a schematic of the cylindrical sample with compression axis vertical). **b** In stage 2, with plastic strain  $>5\%$ , the distribution of stress-drop values is largely unimodal, centered on 25.6 MPa, and is associated with repeated shear on a single dominant shear band (corresponding *inset*). The curves show unimodal and bimodal Gaussian fits of the measured distributions

bands which do not span the entire sample still form from time to time even in this stage) spanning the sample cross-section (*inset b*). As found in the earlier study [18], the stress drops are larger in stage 2.

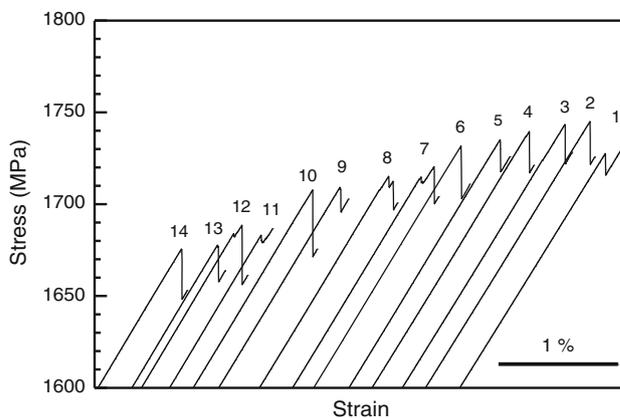
Figure 1b shows the effect of cyclic loading. On each cycle the sample is compressed to the onset of plasticity, and unloading is started as soon as possible after the first stress drop. The maximum and minimum stresses during the brief interval of serrated flow were recorded, and are plotted as series of data points in the figure. It is clear that the flow stress as a function of total plastic strain decreases much faster than that for the monotonically loaded sample. Also shown in the figure is the general trend toward larger stress drops at higher total strain. In effect, the cyclic-loaded sample behaves as though there is no stage 1, only immediate entry into stage 2 behavior.

As the sample geometry influences its deformation behavior the samples of different shape were also tested [21, 22]. Similar behavior on cyclic loading was found for the samples with aspect ratio 1.5:1 and 1:1 (Fig. 3), though the decrease in flow stress as a function of plastic strain is less pronounced in the samples of lower aspect ratio.

Figure 4 shows in close-up the evolution of the yielding behavior of a sample with aspect ratio 1:1 subjected to cyclic loading. On successive cycles, the trend toward earlier yielding is clear, and it is important to understand why this effect arises. SEM (Fig. 5) confirms that though the approach to failure is greatly accelerated, the fracture and cylindrical surfaces demonstrate the same features as

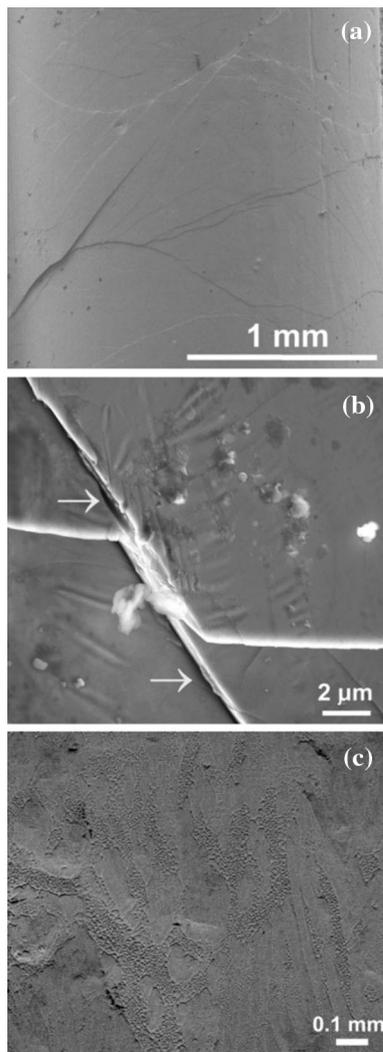


**Fig. 3** Cyclic loading (the stress ratio  $r = 0$ ) of 2-mm-diameter samples with aspect ratio **a** 1.5:1 or **b** 1:1. The data points, labeled with the number of each cycle, give the maximum stress at the onset of yielding, the minimum stress at the end of the stress drop under load, and the value of the stress drop



**Fig. 4** Successive engineering stress–strain curves (close-up of yield onset and initial serrations) obtained on cyclic compression of a BMG sample of 1:1 aspect ratio. Each curve is labeled with the cycle number

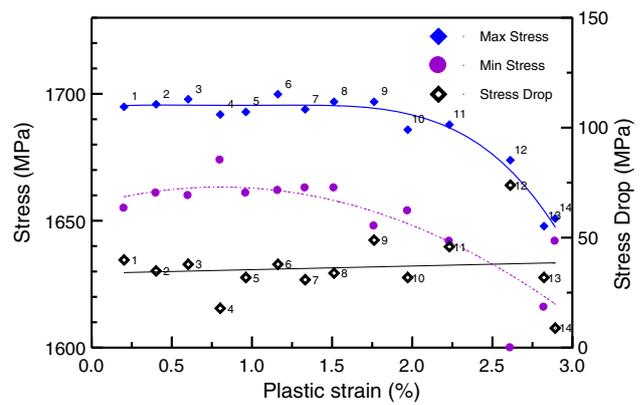
in stage 2 of monotonic loading. Flow starts to be concentrated on a single dominant shear band (Fig. 5a) that leads to micro-cracking (Fig. 5b). Ultimately the sample



**Fig. 5** Scanning electron micrographs: **a** cylindrical surface of a sample with 2:1 aspect ratio after cyclic loading for nine cycles, showing the onset of a dominant shear band; **b** cylindrical surface of a sample with 1.5:1 aspect ratio, showing a micro-crack (*arrowed*); **c** fracture surface of a sample with 1.5:1 aspect ratio after cyclic loading to failure

fails, the fracture surfaces (Fig. 5c) showing the same basic features as for samples that fail under monotonic loading. These features are alternating smooth and vein-patterned areas, associated with variations in crack propagation speed [23].

The cyclic loading tests reported so far have involved decreasing the stress to zero on each cycle. The effects of incomplete unloading (i.e.,  $r = \sigma_0/\sigma_{max} > 0$ ) were also studied. Figure 6 shows the behavior of sample with aspect ratio 2:1 subjected to cyclic loading with  $r \approx 0.12$  studied in comparison with Fig. 1b. Incomplete unloading delays the decrease in flow stress (i.e., delays the onset of stage 2). Interestingly, however, for these two samples failure still occurs at very similar values of total plastic strain.



**Fig. 6** Cyclic loading of samples with 2:1 aspect ratio: with the stress ratio  $r \approx 0.12$ . The data points, labeled with the number of each cycle, give the maximum stress at the onset of yielding, the minimum stress at the end of the stress drop under load, and the value of the stress drop

### Discussion

The cyclic loading applied in this study gives particularly accelerated failure of BMGs, an extreme case of LCF [24] (though with imperfect periodicity the procedure is not identical to conventional LCF testing). Polycrystalline engineering alloys undergo strain-hardening in LCF testing. In contrast, metallic glasses are known to undergo strain-softening, and (as seen in this study) in simple monotonic compression tests the true flow stress is initially roughly constant. In conventional alloys, LCF cracks nucleate along slip bands, grain boundaries, and twin boundaries [25]. In metallic glasses, plastic deformation at room temperature is localized into shear bands and at larger shear offsets these bands evolve into cracks [26, 27]. This appears to be the failure mode in the present cyclic loading tests.

In monotonic compression tests, the plastic flow of metallic glasses shows two stages [18]. In stage 1, many new shear bands appear; these bands are of limited extent (specifically they do not span the sample cross section) and they are distributed throughout the sample. As the plastic strain increases, there is a transition to stage 2 in which strain is increasingly concentrated in one shear band (or a very few bands) spanning the sample cross section. Stage 2 is the prelude to failure, as the large offset on the dominant shear band leads to the sample shape becoming asymmetric. As a result of the distortion, local tension can develop across the dominant shear band and the two sides may part by fingering of air into the liquid-like zone in the shear band [28]. Inevitably, the transition to stage 2 and failure is stochastic [19], depending for example, on how the dominant shear band is located with respect to the extra constraint at the ends of the sample.

The central question for this study is why failure is so accelerated (plasticity so reduced) by cyclic loading even in a purely compressive regime. Under cyclic loading even the engineering flow stress of the samples always decreases with plastic strain, and this decrease is faster at higher aspect ratio (Figs. 1, 3). The average slope of the decrease in stress as a function of strain is 23, 43, and 50 MPa per percent of strain for the samples with 1:1, 1.5:1, and 2:1 aspect ratio, respectively. It is known that the plastic deformation of BMGs and crystalline alloys is enhanced under the condition of confinement loading with lowering the sample aspect ratio [29, 30]. Thus, this trend is as expected, for at higher aspect ratio there is less geometrical constraint on the development of a dominant shear band. At the same time, the data from the different aspect ratios show the reproducibility of the weakening effect of stress cycling, and that similar effects appear at different loading conditions (e.g., different aspect ratios). Also, as it was recently shown [31] the effect of the excess volume in smaller size geometrically similar samples prevails the effect of the sample geometry. However, why is the development of such a dominant band accelerated by cyclic loading? As noted in connection with Fig. 6, even a small (12 %) reduction in stress ratio significantly delays the onset of stage 2.

In the classic Cottrell–Hull mechanism for fatigue initiation in polycrystalline metals, the strain amplitude determines the rate of development of the extrusions and intrusions on the surface of material owing to active slip planes [32, 33]. It is well known that elastic strain at the onset of plasticity is much higher for metallic glasses than for conventional alloys (a consequence of relatively high flow stress and low Young's modulus [4]). In cyclic loading as performed in this study, the elastic axial strain of the sample varies with an amplitude of roughly  $\pm 1$  %. When a metallic-glass sample deforms plastically under axial compressive load, the localized nature of shear-banding must mean that the stress distribution within the sample is highly non-uniform, with regions of elastic compensation ensuring strain compatibility between the shear bands. After unloading, the average stress in the sample is zero, but a pattern of residual stresses remains, especially because after the beginning of plastic deformation there are not-well-developed shear bands (see plenty of such bands in Fig. 5a) which did not cross the entire sample yet and the sample remains in the locally elastically deformed state even after complete unloading (see the inset in Fig. 2a). Evidence for such a pattern after deformation may be found by such techniques as strain-mapping using digital image correlation [34], or mapping of non-uniform hardness [35]. During unloading, then, there are regions in which the local stresses must change sign and tensions must develop.

In particular, although no tensile stress is applied to the sample in the present tests, the shear bands are likely to experience shear-stress reversal during unloading (elastic push-back from the surrounding matrix). There is mounting evidence that such stress reversal is particularly effective in generating free volume. For example, molecular-dynamics simulation has shown that cyclic loading in shear and tension affects the distribution of atomic-level stresses and leads to increased excess free volume of a metallic glass [36]. Our recent observation [37, 38] of cyclic–elastic-deformation-induced nanocrystallization suggests that even in the apparently elastic range, anelastic effects such as the operation of shear-transformation zones generates significant atomic mobility. Recent torsion tests suggest that anelastic effects can be greatly enhanced by stress reversal [39]. We conclude that stress reversal on unloading is likely to reduce the residual strength in the shear bands just activated. That stress reduction favors concentration of shear in existing bands over creation of new bands, and thus accelerates the transition into stage 2 of deformation and then to failure.

The weakening of shear bands by stress reversal is likely to be exacerbated near the sample surface where greater dilatation is possible (by radial movement). Such weakening may underlie the “chipping” phenomenon in compression–compression fatigue described by Freels et al. [14].

## Conclusions

Monotonic compression of  $\text{Zr}_{62.5}\text{Cu}_{22.5}\text{Fe}_5\text{Al}_{10}$  BMG samples confirms earlier findings that there is extensive plasticity and that the plastic deformation proceeds in two stages. In stage 1, new shear bands develop. In stage 2, strain is increasingly concentrated in a single dominant shear band and failure follows. Cyclic loading tests were conducted on the same samples which were repeatedly loaded to the onset of plastic flow and then unloaded. In these tests at  $r = 0$ , there is immediate entry into stage 2 and accelerated failure: the plastic strain at failure is as low as 2.5 %, compared to values as high as 13 % under monotonic compression. Even a small (12 %) reduction in the cycling loading stress amplitude significantly delays the onset of stage 2 deformation. The effect of cyclic loading is attributed to inhomogeneous distribution of the residual internal stresses in plastically deformed sample and the reversal of stresses on active shear bands during unloading. Such reversal, arising from elastic constraint by the matrix around the bands, weakens the sheared material in the bands, favors more inhomogeneous deformation (fewer shear bands with larger shear offsets) and accelerates failure as shear bands with large offsets evolve into cracks.

These results suggest that BMGs may have intrinsically low resistance to LCF, associated largely with their relatively high elastic strain at the onset of yielding. Shear bands weakened by stress reversal may explain the loss of material (“chipping”) from sample surfaces seen in compression–compression fatigue of BMGs.

**Acknowledgements** This study was supported by the World Premier International Research Center Initiative (WPI), MEXT, Japan and in part by the Ministry of Education and Science of the Russian Federation in the framework of Increase Competitiveness Program of NUST «MISiS» (No K2-2014-013).

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