Estonian Journal of Engineering, 2010, **16**, 3, 243–254 doi: 10.3176/eng.2010.3.04

Using small punch testing method for the analysis of creep behaviour of Al-Al_4C_3 composites

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Received 5 April 2010, in revised form 13 July 2010

Abstract. Mechanical alloying and mechanical attrition are both processes based on the imparting of a severe plastic deformation using high-energy ball mills. The experimental material, dispersion-strengthened aluminium with Al_4C_3 particles, was prepared by mechanical alloying of aluminium powder (<50 µm) with different types of carbon. Creep behaviour of the composite, based on aluminium matrix, reinforced by 4 vol% Al_4C_3, was studied at temperatures from 623 to 723 K by small punch testing with a constant force. The time dependence of the central deflection was registered and the minimum deflection rate was determined. The dependence of this quantity on the applied force can be described by a power function with relatively high value of the power. The dependence can be rationalized by an analysis in terms of the threshold concept. Analytical procedure for comparison of the threshold force in small punch experiments and threshold stress in conventional creep testing are given.

Key words: dispersion-strengthened Al-Al_4C_3 composite, mechanical alloying, creep, small punch, threshold stress.

1. INTRODUCTION

Mechanical alloying is a solid state reaction process, in which a mixture of powder(s) is converted into an alloy by facilitating a series of high-energy collisions in a controlled (usually inert) atmosphere. By mechanical alloying, powder particles are repeatedly deformed, fractured and cold welded [1]. This is in contrast to conventional ball milling, in which powder particles are simply mixed while particle size, shape and density change.

The main difference between alloying and attrition milling is that the former starts from elementary pure powders. The severe plastic deformation, imparted to the powder mixture, causes both low temperature alloying and refining of the particles. High production rate, with the capability to produce suficient laboratory and industrial quantity, is an advantage of this mechanical alloying process. The grain size decrease to the nanometer range leads to a drastic increase of the number of grain boundaries, reaching typical densities of 10^9 interfaces per cm^3. The large concentration of atoms, located in the grain boundaries, in comparison with the crystaline part scales roughly with a reciprocal grain size d dependence as $3\delta/d$, δ being the width of the interface, typically 1.2 nm [2]. For example, the stored enthalpy reaches values up to 10 kJ/mole for Ru after 32 h milling, which corresponds to the heat of fusion fraction ΔH_f up to 30–40%. These energy levels cannot be achieved by incorporation of defects, which are found during conventional processing (cold rolling, extrusion etc.). The maximum dislocation densities, which can be reached in intensively deformed metals, are less than $10^{16}/m^2$, which corresponds to energy of less than 1 kJ/mole. Therefore, it is assumed that the major energy contribution is stored in grain boundaries and related strains within the nanocrystalline grains, which are induced through grain boundary stresses [2–4].

It has been accepted that both cold welding and fracture, which distinguish mechanical alloying from conventional ball milling, happened during the process. All the time steel balls collide with the internal mill wall, powder particles are trapped. The impact force deforms the particles and creates new atomically clean surfaces [5–7]. Thus cold welding can occur between two such surfaces when they are compressed into contact during subsequent collisions. Solid state reactions can then occur across these new, internal interfaces, allowing a change of the chemical composition of the particles progressively during milling [8].

The dispersion-strengthened alloys Al-Al_4C_3, prepared by mechanical alloying using powder metallurgy technology, are promising structural materials enabling significant cuts of weight for use mainly in aircraft and car industry, also at elevated temperatures [9–11].

The small punch (SP) technique represents one of recent methods for estimation of mechanical properties of solids [12,13]. The method uses small disc specimens up to 10 mm in diameter and thickness up to 0.5 mm. The specimen, placed on the ring (clamped eventually at its edges) and a ball (or a punch with hemispherical tip) is forced into the centre of the specimen either at constant velocity or at constant force. Load vs displacement curve is registered in the former test type that is analogical to a stress vs strain test under a constant strain rate. In the latter test, the time dependence on the punch displacement, i.e. the deflection in the centre of the disc specimen is recorded. This variant is similar to conventional constant load creep tests and it will be the subject of the present paper. The main advantages stem from miniaturized specimen size: the necessary volume is small and the technique thus enables testing of the material removed from inspected construction parts without affecting functionality of these parts and the specimens can be extracted from critical regions of heterogeneous materials that are smaller than dimensions of specimens for conventional tests.

2. EXPERIMENTAL MATERIAL

Experimental material was prepared using powder metallurgy route by mechanical alloying. Aluminium powder of particle size of <50 μm was dry milled in an attritor for 90 min with the addition of graphite KS 2.5 in the amount corresponding to 4 vol% of Al_4C_3 in the resulting product. The mixture was then cold pressed using a load of 600 MPa into cylindrical shape compacts. Subsequent heat treatment at 823 K for 3 h induced chemical reaction $4Al + 3C = Al_4C_3$. The cylinders were then hot extruded into rods at 873 K with 94% reduction of the cross-section. Due to a high affinity of Al, the residual porosity of this material was less than 1 vol%. The experimental material was produced in the Institute for Chemical Technology of Inorganic Materials, Technical University of Vienna.

When describing microstructures, one has to consider geometrical and morphological factors. According to the microstructure observations, the particles in our materials can be divided into three distinctive groups: A – small Al_4C_3 particles, identified by TEM on thin foils, with mean size approximately 30 nm, which made up to 70% of the dispersoid volume fraction; B – large Al_4C_3 particles with mean size between 0.4 and 2 μm, found on SEM metallographic micrographs; and C – large Al_2O_3 particles with mean size of 1 μm. Al_4C_3 particles are elongated and Al_2O_3 are spherical. The grain size of this material was about 380 nm. Substructures of the initial Al-Al_4C_3 material with 4 vol% of Al_4C_3 are shown in Fig. 1 (transverse direction) and Fig. 2 (longitudinal direction).

Particles of all categories during the high plastic deformation are distributed in rows. The particles are spherical or have only a low aspect ratio, so that they can be considered as spherical.

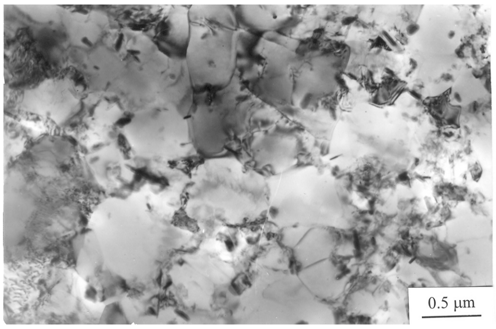

Fig. 1. Substructure of initial Al-Al_4C_3 material with 4 vol% of Al_4C_3 (transverse direction).

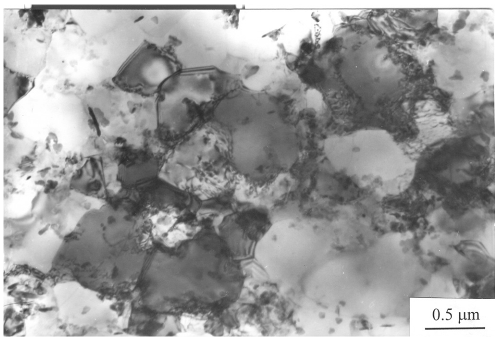

Fig. 2. Substructure of initial Al-Al_4C_3 material with 4 vol% of Al_4C_3 (longitudinal direction).

2.1. Test method

The specimens for small punch testing were prepared by cutting slices 1.2 mm thick and of 8 mm diameter using spark erosion. The slices were perpendicular to the extrusion axis. The slices were ground carefully from both sides equally and finally polished to 1200 grit. The final thickness of 0.500 ± 0.002 mm was measured by a micrometer with a resolution of 1 μm.

A simple schematic drawing of the small punch testing assembly is given in Fig. 3. The disc specimen is placed on the ring (lower die) with inner diameter 4 mm. The specimen is clamped by an upper die in such a manner, that the upper die prevents an upward bending of the specimen during its deformation. No effort is done to prevent radial displacement of specimen matter. The specimen holder is then placed into the creep machine.

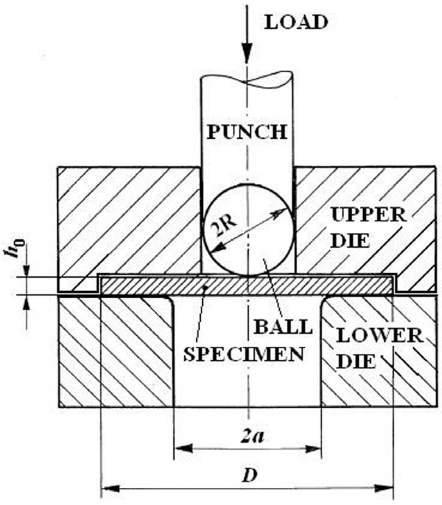

Fig. 3. Schematic drawing of the experimental setup.

The constant load cantilever creep machine was adapted for SP testing. The machine enables testing at temperatures up to 1173 K with forces from 20 to 7000 N. The tests are performed in protective argon atmosphere. During the test, a precise ceramic ball made of FRIALIT F99.7, 2.5 mm in diameter, is pushed with a constant force against the specimen. Central deflection is measured as the difference in the positions of the punch and lower die, using a linear variable differential transformer W2K from Hottinger-Baldwin Co. (Germany) and is continuously recorded with a PC.

3. EXPERIMENTAL RESULTS AND DISCUSSION

An example of the dependence of the central deflection on time, obtained in the small punch test arrangement, is given in Fig. 4. It can be seen that the same general feature of the curve can be observed as in conventional creep tests. The detected curve has a pronounced primary stage in which the deflection rate gradually decreases. The steady state stage is apparently missing but the minimum deflection rate can be evaluated. After reaching the minimum, the deflection rate is steadily increasing until disc fracture occurs.

The dependence of the minimum deflection rate on the applied force for two temperature levels is given in Fig. 5 in bilogarithmic coordinates. The dependence can be described by the power-law relationship of the form

$$\dot{\delta}_{\mathrm{M}} = A_{\mathrm{S}} F^{n_{\mathrm{S}}}, \tag{1}$$

where $\dot{\delta}_{\mathrm{M}}$ is the minimum deflection rate, F is the acting force and A_{S} is a constant characterizing the temperature dependence. The exponent n_{S} values are

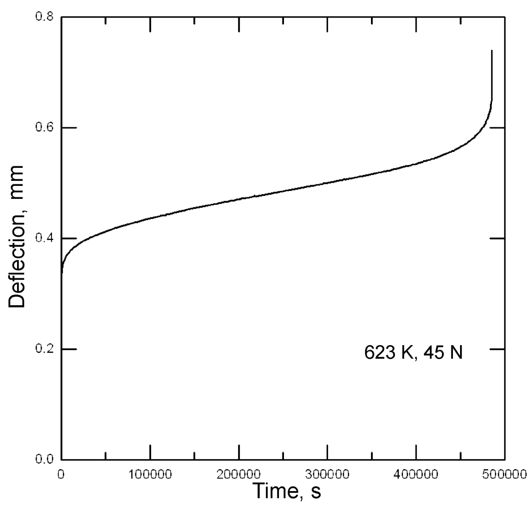

Fig. 4. Example of time dependence of measured central deflection.

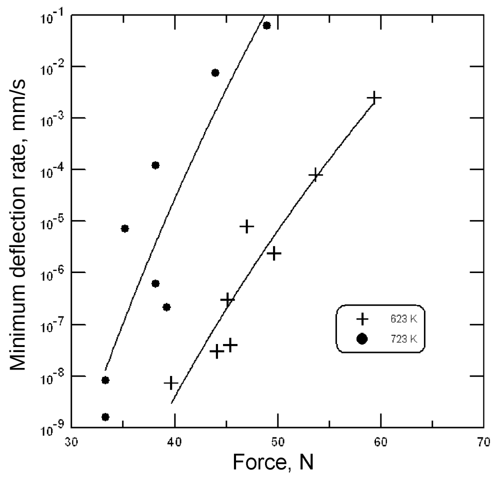

Fig. 5. Dependence of the minimum deflection rate on the applied force.

33.29 at 623 K and 41.56 at 723 K, respectively. These values are slightly greater than the values of stress exponents of minimum creep rate in tension determined for a similar Al composite (21.5 at 623 K and 21.4 at 723 K, respectively [14]).

The dependence of the time to fracture, t_F, on the applied force F is given in Fig. 6. The dependence can also be described by a power law of the type of Eq. (1), i.e.

$$t_F = A_F F^{n_F}, \tag{2}$$

with negative values of the power $n_F = -36.59$ at 623 K and −32.71 at 723 K. These values can again be compared with values of stress exponents of time to

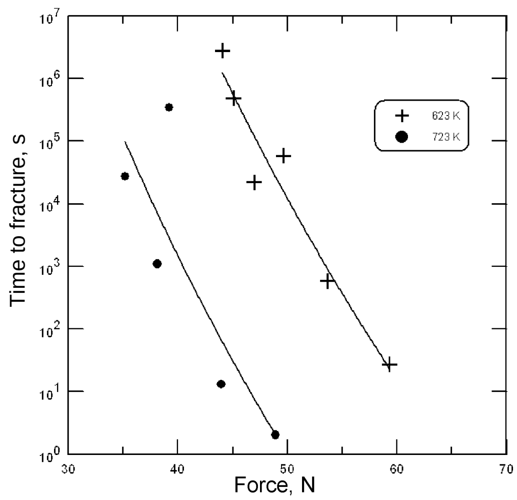

Fig. 6. Dependence of time to fracture on applied force.

fracture in conventional tensile creep tests (–18.1 at 623 K and –18.9 at 723 K, respectively [14]).

Time to fracture, t_F, and minimum creep rate, $\dot{\varepsilon}_M$, in uniaxial creep tests are related by the well-known Monkman–Grant equation [15]:

$$\log t_F + m_C \log \dot{\varepsilon}_M = C_C, \tag{3}$$

where m_C and C_C are constants. A similar equation also applies for the small punch test data,

$$\log t_F + m_S \log \dot{\delta}_M = C_S, \tag{4}$$

with constants m_S and C_S. This is demonstrated in Fig. 7. The value of constant m_S is close to 1 (1.057 at 623 K and 0.989 at 723 K).

The deflections observed at fracture in small punch tests, δ_F, for both temperatures are given in Fig. 8. These deflections are lower than fracture deflections found in heat-resistant steels [16,17]. This fact corresponds to low creep ductility of this composite [14]. The values of the initial deflection are given for comparison.

The values of force exponent n_S (as well as the values of stress exponent in the similar composite [14]), given in the previous paragraph, are very high by comparison with the stress exponents reported for the creep of pure aluminium. Such high values are usually explained by means of the threshold stress concept (one of the latest reviews can be found in [18]). From a detailed inspection of Fig. 5 it follows that the existence of a threshold force – analogical to the threshold stress in conventional creep experiments – can be indicated also in the present SP results. This can be proved by plotting $(\dot{\delta}_M)^{1/n}$ against the applied force F on a linear axis for n selected values, corresponding to creep in pure matrix. The value $n = 5$ was chosen in agreement with Orlová et al. [14]. The

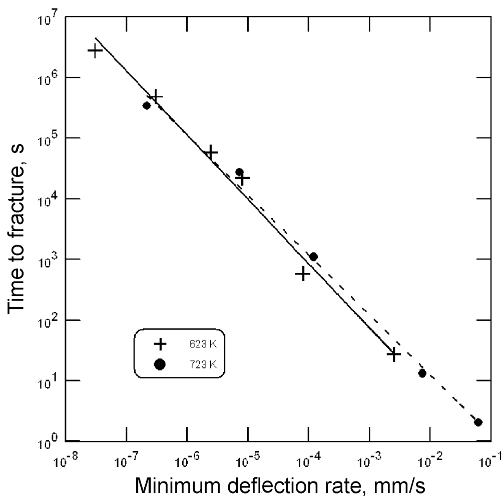

Fig. 7. Dependence of the time to fracture on the minimum deflection rate.

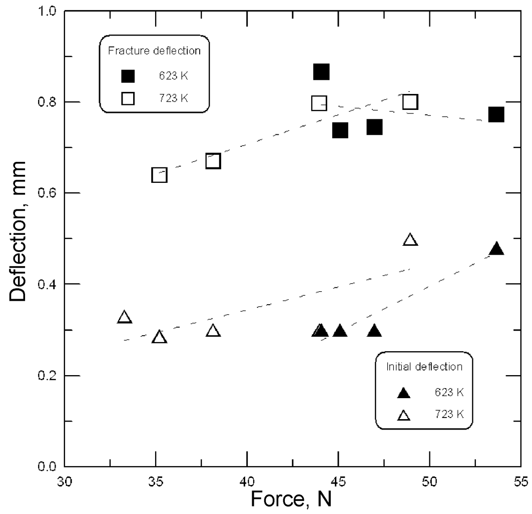

Fig. 8. Force dependence on the initial deflection and fracture deflection.

intercept with the x-axis is equal to the threshold force F_{th} (cf. Fig. 9). The force dependence of the minimum deflection rate can thus be described by the equation

$$\dot{\delta}_{\mathrm{M}} = A(F - F_{\mathrm{th}})^5. \tag{5}$$

An inclusion of the threshold force into the analysis of results of SP tests thus reveals dependences comparable with those observed in conventional creep and predicted by theoretical models. The estimated values of the threshold force together with the threshold stress are given in Table 1.

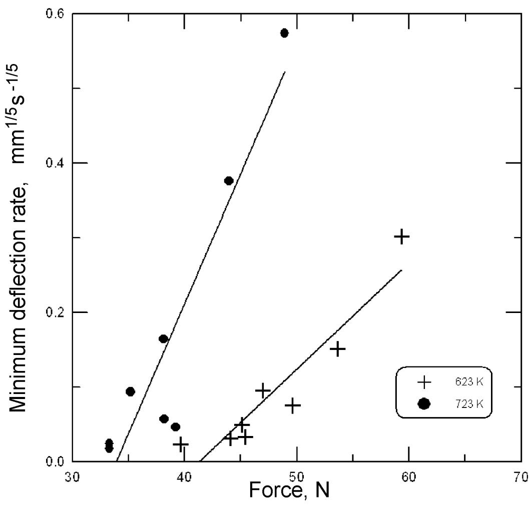

Fig. 9. Relation between $\dot{\delta}^{1/n}$ and force for the true exponent $n = 5$.

Table 1. The estimated values of the threshold force together with the threshold stress

Temperature, K	Threshold force, N	Threshold stress, MPa	$\frac{F_{th}}{\sigma_{th}}$, N/MPa
623	41.3	51.9	0.79
723	33.9	46.6	0.73

An important problem of the SP test is the relation of its quantities, e.g. force or deflection rate, to the quantities recorded during the uniaxial creep test, i.e. stress and creep rate. The test is complex and numerical procedures like finite element modelling are required [19–21]. We would like to demonstrate problems, encountered by analytical approaches, with a relatively simple relation of threshold stress and threshold force. Assuming that the friction forces in contact area between the punch and the specimen prevent straining, the maximum stress at the perimeter of this area [22] is

$$\sigma_{max} = \frac{F}{2\pi Rh\sin^2\alpha}, \tag{6}$$

where F is the acting force, R is the punch radius, h is the actual thickness of the specimen and $\tan\alpha$ is the local slope at the perimeter of contact area. If $\sigma_{max} \le \sigma_{th}$, the specimen cannot creep. The threshold force is then given by the equation

$$\frac{F_{th}}{\sigma_{th}} = 2\pi Rh\sin^2\alpha. \tag{7}$$

The contact angle α can be calculated from the geometry of the specimen during the test using Timoshenko's equation [23,24] for the deflection of a centrally loaded circular plate. The contact angle α is then related to the central deflection δ by (Fig. 10):

$$\frac{\delta}{R} = 1 - 0.5\cos\alpha - \frac{1}{2\cos\alpha} - \frac{\left(\frac{a}{R}\right)^2 - \sin^2\alpha}{4\cos\alpha\ln\frac{R\sin\alpha}{a}}. \tag{8}$$

The mean actual thickness of the specimen can be approximated as

$$h = h_0\cos\alpha. \tag{9}$$

This approximation assumes constancy of the specimen volume, i.e. significant cracking does not occur. The real value of thickness at the perimeter of the contact area is certainly smaller than the average value. Thus the thickness

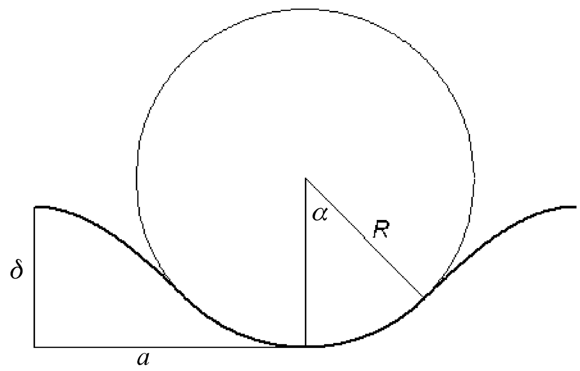

Fig. 10. Schematic illustration of the specimen geometry during the small punch test.

given by Eq. (9) has to be considered as an upper bound. The dependence of the force F on central deflection δ calculated by means of Eqs. (7) to (9) is given in Fig. 9. Good agreement between calculated and experimentally estimated values of the ratio is obtained for fracture deflections that are in the range from 0.6 to 0.8 mm (Fig. 11).

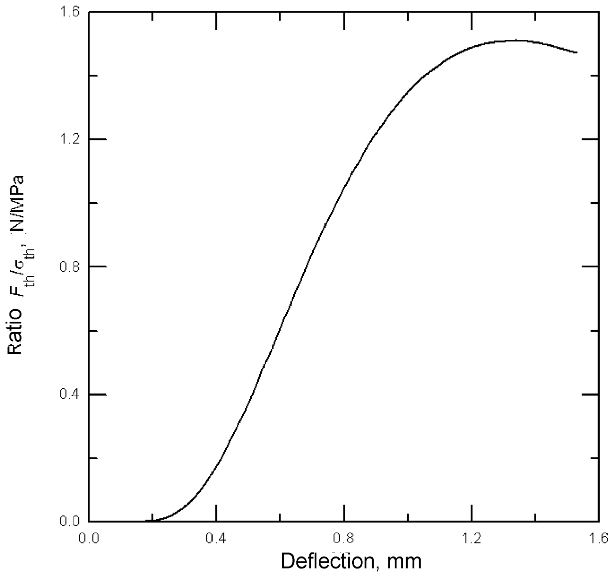

Fig. 11. Dependence of the calculated ratio of the threshold force on the threshold stress by central deflection.

4. CONCLUSIONS

1. The small punch tests of the mechanically alloyed Al composite were performed in constant force regime at elevated temperatures. The force dependences on the minimum deflection rate and on the time to fracture are comparable with the analogical dependencies of conventional uniaxial creep tests of the similar alloy.
2. The force dependence on the minimum deflection rate can be described in terms of the threshold stress/threshold force concept.
3. Relation between the threshold force in the small punch test and the threshold stress in uniaxial creep test is presented. This is based on an analytical expression for the maximum stress in the contact-free region of the disc specimen.

ACKNOWLEDGEMENT

Part of this work was carried out with support of the Science Grant Agency of Slovakia under the projects VEGA 2/0105/08.

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Indentimismeetodi kasutamine Al-Al_4C_3 komposiitide roome uurimisel

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Mehaaniline legeerimine ja jahvatamine kõrgenergeetilistes kuulveskites on protsessid, mis põhinevad korduval plastsel deformatsioonil. Selle kirjeldamiseks on kasutatud indentimist. Eksperimentaalmaterjal – Al_4C_3 osakestega dispersioontugevdatud alumiinium – valmistati alumiiniumpulbri mehaanilise legeerimise teel süsinikuga. Määrati Al-maatriksi ja Al_4C_3 osakeste Martensi kõvadus HM, indentimismoodul E_{1T} ning deformatsioonitöö W, kasutades pinnatundlikku indentimistehnikat. Selgus, et Al_4C_3 osakeste kõvadus on 5–7 korda suurem kui Al-maatriksil. Uuriti alumiiniummaatrikskomposiidi (4 mahuprotsenti Al_4C_3) roomavust jäävkoormusel temperatuuridel 623 kuni 723 K, kasutades indentimiskatset. Registreeriti tsentraalse läbipainde sõltuvus ajast ja määrati minimaalne läbipainde kiirus. Nimetatud näitajat on võimalik kirjeldada suure astmenäitajaga astmefunktsiooniga. Sõltuvust on võimalik lihtsustada lävekontseptsiooni kasutades. On kirjeldatud jõuläve ja sellele vastava pingeläve analüüsi protseduuri indentimise tavaroomel.