

SELECTED PROPERTIES OF MULTILAYER FILMS APPLIED FOR VACUUM AND MODIFIED ATMOSPHERE PACKAGING SYSTEMS

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ABSTRACT

The aim of the research was to determine the selected properties of packaging materials applicable for vacuum and modified atmosphere packaging. Six samples of multilayer films with different composition were tested to evaluate the thickness, basic weight, density and the mechanical properties in elongation and puncture tests. Zwick/Roell apparatus equipped with elongation jaws and a puncture pin was used in the experiments. The tensile modulus, tensile strength, elongation at tensile strength, stress at break and work at break were investigated with the elongation test, whereas the breaking force, puncture work and extension at break were evaluated with the puncture test. The obtained results allow conclusion that the thickness and basic weight of the packaging films used in the vacuum system was higher comparing with the MAP system. Analyzing the results of the elongation modulus and stress at break it was reported that the materials used for food packaging in the MAP system were characterized with better properties, whereas elongation at the tensile strength was higher for films applied in the vacuum system, except for a film with a metalized layer. The highest resistance for puncture was evaluated for OPET/PE film used in MAP packaging. The extension at break under the puncture test was higher for films dedicated for vacuum packaging suggesting their better resistance for the mechanical damage by a thin pin. Several correlations between physical and mechanical properties of multilayer films were found.

Introduction

Problems of quality features, including strength properties of materials used for packaging of food products, are very important both for the food industry, catering of ready meals, transport and storage, and for the common consumers for many reasons (Olech and Kuboń, 2016). Packaging made of elastic films must have an appropriate mechanical and barrier properties, and thermal strength. These features have a significant impact on the selection of packaging materials for various groups of products and packaging systems.

The production of plastics is constantly growing. In 2005 the world production was 230 million tons, while in 2015 – 322 million tons. The popularity of plastics is due to the pos-

sibility of using them in various fields (Maeda et al., 2015). Plastic packaging materials can generate great environmental problems, but if the proper recycling standards and methods will be applied, it is possible to avoid the pollution or reuse the plastic wastes for other applications, not only in food packaging (as a secondary package) but also for many industrial applications, as for horticulture, automobile, construction or insulation materials. The packaging industry is using a wide range of polymers to obtain elastic films and rigid packaging forms with high, repeatable and stable quality parameters for various applications. Compliance for the creation of various forms, flexibility, stiffness, differentiated barrier (for oxygen, water vapor or light), chemical resistance, ease of sealing and welding, low weight and many other features determined the usefulness of polymer films in new packaging methods (Panfil-Kuncewicz et al., 2011).

Modern techniques were implemented to improve the food storage time and quality, including aseptic packaging, vacuum packaging and modified atmosphere packaging (MAP). An important impulse for these changes was the widespread distribution of food products in modern transportation and storage systems, as well as the sale of unit packages with an extended shelf life in shops and supermarkets. From several packaging methods, the most useful are vacuum and MAP techniques. The main tasks of vacuum packaging include protection of products against development of microorganisms, extending the shelf life of the packaged product, limiting drying, loss of taste and smell, protection against touch and mechanical damage, reduction of mass losses and elimination of the influence of environmental factors, such as moisture or oxygen. Vacuum packaging consists completely in removing the air out of the package, after which it is hermetically sealed by welding. Vacuum packaging due to the use of transparent foils enables attractive and hygienic presentation of goods. The tightness of the sealing and appropriate barrier features are important advantages to protect against loss of freshness and extend the shelf life of products up to 5 times longer than those stored under normal conditions (Brennan and Day, 2006). Vacuum packed goods include fresh meat and meat products, cheese, dairy products, sterilized ready meals and industrial and medical articles (Kacanak et al., 2005). This process is carried out with flexible materials that have the ability to shrink.

The MAP system is a type of packaging in which the so-called protective atmosphere is used instead of air. The protective atmosphere is a mixture of gases such as oxygen O_2 , nitrogen N_2 , and carbon dioxide CO_2 . In the case of vacuum packaging, removal of oxygen may create favorable conditions for the development of anaerobic microflora and contribute to the destruction of the texture of delicate products. The packaging of food in a modified atmosphere eliminates these drawbacks and additionally, the introduction of a properly selected gas mixture for a specific type of products will have bacteriostatic and bactericidal effects on many types of microbes' undesirable in food, which results in a prolonged freshness of packaged products (Guillard et al., 2010). The process of packaging food in a modified atmosphere is based on the evacuation of air and then completely or partially re-filling the package with an appropriate gas mixture. The next step is hermetic sealing of the package. It is important to use high barrier materials for MAP packaging, which will ensure keeping the optimized atmosphere inside the package.

The packaging of food products uses many types of flexible films which are the most frequently chosen by producers due to the low cost of production, desired properties and continuous development of production technology along with innovation (Miroslaw, 2010). The films used for packages formation are made with plain foils, homogeneous foils and

multilayer laminates. Homogeneous films have several layers, and are made of the same or similar composition of polymers, for example PE-LD and PE-LLD. Multilayer films or laminates consist of several layers and they compose of different polymers. They are manufactured by co-extrusion or with various lamination techniques (Czerniawski and Michniewicz, 1998). In a multilayer material, at least one layer should be made with thermoplastic polymer that enables sealing by a hot welding process. The most popular polymers used in the flexible film processing are polyethylene (PE), polypropylene (PP), polyamide (PA) and polyethylene terephthalate (PET). Polyethylene is a plastic material belonging to a group of polymers called polyolefins. It is usually categorized by its density as indicated in the abbreviations e.g. PE-HD (high density PE), PE-MD (medium density PE), PE-LD (low density PE), PE-LLD (linear low density PE). Polyester PET films are used as one of the most important components for many barrier laminates, also for that intended for pasteurization or sterilization together with the product due to the high heat resistance (Sharon and Sharon, 2012). Polyethylene terephthalate polyester (PETP), OPET – oriented version of PET or PETX - PVDC coated PET, can be applied as a thin-film coating for the effective improvement of the barrier properties in the composition of flexible films with high barrier requirements (Maeda et al., 2015). The introduction of ethylene vinyl alcohol (EVOH) layer gives the material very high limitation of oxygen and water vapor transmission levels. Excellent barrier properties of the EVOH copolymer (about 10000 times better than PE layer of the same thickness) protecting against penetration of oxygen and moisture, guarantee safety and are excellent in the packaging of food, medical, pharmaceutical products, cosmetics as well as agricultural and industrial products (Butler and Morris, 2013). Metalized layer (MET) is usually used to protect products from UV light and improve the barrier properties of a coated film. These polymers are used as components of multilayer materials for the MAP or vacuum packaging due to high barrier properties.

It is important that the quality of the packaging materials provides the best possible protection against the influence of undesirable external factors. Therefore, mechanical properties, such as for example puncture resistance or tensile strength play the most important role in this issue.

Objective, scope and methods of research

The objective of the paper was to evaluate the selected properties of films used in the vacuum and in modified atmosphere packaging systems. The thickness, basic weight, density, tensile at elongation and puncture resistance of various types of films were determined as selected properties. The study involved 6 different types of flexible films provided by MULTIVAC Sp. z o. o. (Natalin, Poland). The first 3 foils (PA/PE, PETP/MET/PE, PA/EVOH/PE) were commercially used in vacuum packaging, while the next 3 foils (PETX/PE, PET/PA/EVOH/PE, OPET/PE) were the films used in MAP systems. Ten samples of each film were prepared.

The thickness was measured with a micrometer screw with accuracy of 0.0005 mm in 10 replications for each sample. The basic weight of the tested film samples was determined on a laboratory scale with the accuracy of 0.001 g. 15 samples with determined dimensions were weighed on the scale. The basic weight ($\text{g}\cdot\text{m}^{-2}$) of the tested films was de-

terminated as the mass of the sample to the sample surface. Film density ($\text{g}\cdot\text{cm}^{-3}$) was calculated as the mass of volume of the tested material (Rejak et al., 2013).

The testing of the mechanical properties of packaging films was done using the Zwick/Roell BDO-FB0.5TH (Zwick GmbH & Co., Ulm, Germany) testing machine with specific attachments, according to ASTM D882 and ASTM D63805 Standards. Specimen shape should be rectangular stripes, as required by ASTM D882: 10 samples of each film type with dimensions of 20x200 mm were cut along the packaging materials for the tests. The testing machine was equipped with stretching jaws attachments to evaluate the tensile properties and with Parker pen pin with a diameter of 0.8 mm for the puncture test. The test speed was set at 100 mm min^{-1} until the sample was broken. For the elongation test, the distance between the jaws at the starting position was set at 100 mm. During the tensile test, the following parameters were determined: tensile modulus (MPa), tensile strength (N), elongation at tensile strength (%), stress at break (MPa), and work at break (J) (Rejak et al., 2013). For the puncture test, according to DIN EN 14477, prepared samples of a film 20 mm wide were placed in the test fixture and clamped in such a way that the pin was directly above the opening. The pin travel speed was 100 mm min^{-1} , the distance of the handles at the starting position was 5 mm. The following parameters were recorded during the puncture test: breaking force (N), puncture work (mJ), and extension at break (mm) (Chocyk et al., 2015). The measurements were made in 10 replications for each type of the tested film, taking as the result the average of the measurements.

Statistical analysis was conducted using Statistica software version 13.3 (Dell Inc., StatSoft, Poland). The results were evaluated for homogenous groups using the ANOVA analysis of variance and the Pearson correlation coefficients have been found between all the tested features at a level of significance $\alpha=0.05$.

Results and analysis

Thickness measurements of films used in vacuum and MAP packaging showed two groups of results for tested materials (Table 1). The lowest thickness was determined for multilayer PETX/PE film, but also the low thickness was observed for PET/PA/EVOH/PE and PETP/MET/PE with an aluminum layer with insignificant differences between the samples. Similar thickness was observed for OPET/PE and PA/PE films. The PA/EVOH/PE film was characterized by almost twofold thickness compared to other materials. The thickness of the tested films was highly correlated with the basic weight, where similar significance of differences was observed.

The basic weight of the tested films varied from $64.26 \text{ g}\cdot\text{m}^{-2}$ for PETX/PE film used in MAP systems up to $143.32 \text{ g}\cdot\text{m}^{-2}$ for PA/EVOH/PE multilayer film dedicated for vacuum packaging. Generally, the thickness and basic weight of films useful for vacuum packaging were greater due to the technological requirements for packaging materials as the resistance to mechanical damage, shrinking ability, resistance to oxygen permeability, but also there are lower requirements for transparency of films for vacuum packaging, so they can be much thicker than for the MAP system. The results of density (Table 1) showed lower density of films for vacuum packaging than for packaging in a modified atmosphere, significantly higher density was found for OPET/PE film due to orientation of PET film during processing. The density of pure PE is ranging between $0.917\text{-}0.930 \text{ g}\cdot\text{cm}^{-3}$ (Mosleh et al.,

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1998). Application of various layers in multilayer films may affect the density of plastic films. The highest density was evaluated for OPET/PE composite material applicable in the MAP system, and the lowest density was calculated for PA/EVOH/PE film. Similar results were found for PA/PE film used for vacuum packaging. However, no direct correlation of the density with thickness and basic weight results was observed.

Table 1.
Thickness, basic weight and density of tested vacuum and MAP films

Packaging method	Film type	Thickness (mm)	Basic weight ($\text{g}\cdot\text{m}^{-2}$)	Density ($\text{g}\cdot\text{cm}^{-3}$)
Vacuum	PA/PE	0.0880 ^{ab} ± 0.002	82.184 ^{ab} ± 1.37	0.9339 ^a ± 0.015
	PETP/MET/PE	0.0750 ^a ± 0.000	73.368 ^a ± 2.05	0.9782 ^{ab} ± 0.027
	PA/EVOH/PE	0.1605 ^b ± 0.001	143.316 ^b ± 5.85	0.8929 ^a ± 0.036
MAP	PETX/PE	0.0650 ^a ± 0.000	64.263 ^a ± 2.28	0.9886 ^{ab} ± 0.035
	PET/PA/EVOH/PE	0.0675 ^a ± 0.025	64.631 ^a ± 2.07	0.9575 ^{ab} ± 0.031
	OPET/PE	0.0900 ^{ab} ± 0.000	100.658 ^{ab} ± 3.08	1.1184 ^b ± 0.034

^{a-b} means with the same letters in columns not differ significantly at $\alpha=0.05$; n=10

Polyethylene itself has a low strength, hardness and rigidity, but it has a high extensibility and impact strength (Sazali et al., 2010). Application of other polymers as components of multilayer films may improve the tensile strength but also decrease the elasticity and elongation (Davis, 2004). Typical PE-LD tensile elongation is 600%, whereas PE-HD is 900% (Mosleh et al., 1998). The tensile test measures how strong is the material (the tensile strength), how flexible it is (the elongation), and how stiff it is (the tensile modulus). The results of the elongation test of films applicable for vacuum and MAP packaging are presented in Table 2.

Table 2.
Results of mechanical properties of tested vacuum and MAP films under the elongation test

Packaging method	Film type	Tensile modulus (MPa)	Tensile strength (N)	Elongation at tensile strength (%)	Stress at break (MPa)	Work at break (J)
Vacuum	PA/PE	235 ^a ± 14.9	47.6 ^a ± 3.7	300 ^c ± 17.0	26.9 ^a ± 2.0	11.3 ^b ± 1.1
	PETP/MET/PE	464 ^b ± 7.2	46.4 ^a ± 0.9	37 ^a ± 2.6	24.0 ^a ± 1.5	1.8 ^a ± 0.1
	PA/EVOH/PE	161 ^a ± 5.1	84.5 ^b ± 6.3	410 ^d ± 19.0	26.4 ^a ± 1.9	24.8 ^c ± 2.4
MAP	PETX/PE	520 ^b ± 10.6	58.1 ^a ± 2.0	70 ^b ± 6.7	44.7 ^b ± 1.6	3.8 ^a ± 0.4
	PET/PA/EVOH/PE	689 ^{ab} ± 27.6	55.4 ^a ± 2.4	44 ^a ± 9.7	35.0 ^{ab} ± 4.5	2.7 ^a ± 0.6
	OPET/PE	1000 ^c ± 16.1	124.3 ^c ± 3.4	81 ^b ± 7.1	68.6 ^c ± 1.9	10.6 ^b ± 1.0

^{a-c} means with the same letters in columns not differ significantly at $\alpha=0.05$; n=10

The results of the tensile modulus of films applicable for the vacuum packaging systems showed that the obtained values were significantly lower than the results of films used for food packaging in MAP systems. The values of the tensile modulus ranged from 161 MPa for PA/EVOH/PE film, 235 MPa for PA/PE film up to 464 MPa for PETP/MET/PE film containing a metalized layer in the composition (Table 2). The tensile strength results did

not show similar tendencies, correlation between these tensile properties were insignificant (Table 4). Analyzing the results of the tensile modulus determined during the testing of films used for packaging in the MAP system it was found that OPET/PE film was characterized by a significantly higher tensile modulus (1000 MPa) and tensile strength (124.3 N) in relation to the remaining samples (520-689 MPa for tensile modulus, and 55.4-58.1 N for tensile strength, respectively). It could be due to the significant and high correlation of tensile modulus ($r=0.908$) and tensile strength ($r=0.65$) with the density of the tested films (Table 4). The tensile modulus results were highly correlated with elongation at tensile strength and stress at break of the tested films. The elongation at tensile strength was the highest for PA/EVOH/PE film dedicated for vacuum packaging. Similar results were found for PA/PE film (Table 2). Compared to pure PE elongation properties (Mosleh et al., 1998) the addition of other than PE polymers, incorporated into multilayer materials, lowered the elongation properties of the tested films. It was noticed that the elongation at the tensile strength of PETP/MET/PE film used for packaging in a vacuum system showed a value similar to the elongation at the tensile strength of films used in food packaging in the MAP system, which mostly contains oriented films with less elasticity within their composition. The lowest elongation was observed for the film with metalized layer comprised in the structure which is resulting in diminishing elasticity of the film with values 10 times lower than for the previously mentioned ones. Except of PETP/MET/PE the lower elongation was noted for the MAP system films ranged from 44 to 81%. The results of elongation at the tensile strength were significantly correlated with thickness, basic weight, density, as well as with tensile modulus (Table 4). During the elongation test of films, the stress at break of the films used for the vacuum packaging system was significantly lower than for the MAP technique, but the results of work at break were much higher, except of the metalized film (Table 2). The results of stress at break of the packaging films showed that materials used for packaging in the MAP system reached higher values than films used for vacuum packaging. The stress at break values for films used in the vacuum system were similar with no significant differences at 0.05 (Table 2). The stress at break ranged from 24.0 to 26.9 MPa. The highest value of the stress at break, 68.6 MPa, was determined for OPET/PE film used in the MAP system. The results of the work needed to destroy the tested films present the higher work at the break for films used in vacuum packaging than for films used for packaging in the MAP system. Among the values obtained during the elongation of films used for packaging in a modified atmosphere, the highest work at break 10.6 J was also recorded for OPET/PE film. Work at break values during the testing of PA/EVOH/PE film were the highest (mean 24.8 J) of all packaging materials tested. Stress at break results were significantly correlated with the density and tensile characteristics whereas the work at break mostly depend on the film thickness, basic weight and elongation with high correlation coefficients of 0.966, 0.959 and 0.909, respectively (Table 4).

During the test of resistance to puncture the packaging materials, selected parameters were determined as the breaking force, puncture work and extension at break. The breaking force indicates the resistance of the material to destruction by the sudden impact with the pin, which is particularly important when packaging materials are used in modern techniques as MAP and vacuum packaging. The higher the breaking force the better protection of the product from damage and changing the atmosphere inside the package. The breaking force tests in the puncture test were carried out for both films used for food packaging in the MAP and in the vacuum packaging system. The results are presented in Table 3.

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Table 3.
Results of mechanical properties of tested vacuum and MAP films under the puncture test

Packaging method	Film type	Breaking force (N)	Puncture work (mJ)	Extension at break (mm)
Vacuum	PA/PE	4.1 ^a ± 0.1	6.0 ^a ± 0.6	2.6 ^b ± 0.1
	PETP/MET/PE	8.1 ^b ± 0.3	7.1 ^a ± 0.8	2.3 ^{ab} ± 0.1
	PA/EVOH/PE	6.6 ^{ab} ± 0.2	10.1 ^{ab} ± 1.2	2.8 ^b ± 0.1
MAP	PETX/PE	6.8 ^b ± 0.5	5.4 ^a ± 1.1	2.1 ^a ± 0.1
	PET/PA/EVOH/PE	7.0 ^b ± 0.3	5.7 ^a ± 2.3	1.8 ^a ± 0.4
	OPET/PE	15.4 ^c ± 0.7	12.2 ^b ± 1.4	1.9 ^a ± 0.1

^{a-b} means with the same letters in columns not differ significantly at $\alpha=0.05$; n=10

Among the tested materials, the highest value of the breaking force was obtained during the OPET/PE film test used in the MAP packaging system with the value of 15.3 N. This is about 3-times higher than the breaking force value obtained during the measurement of PA/PE film (4.1 N). The content of polyethylene terephthalate (PET) like PETP, PETX or OPET in films composition significantly improved the resistance of packaging materials to destruction during the puncture test. The breaking force determined during the puncture of the films used in vacuum packaging assumes the values from 4.1 to 8.1 N, while for MAP packaging materials values were from 6.8 to 15.4 N. The breaking force results were significantly correlated with the density, tensile modulus, tensile strength and stress at break with coefficients of 0.905, 0.846, 0.864, and 0.853, respectively. The results of puncture work necessary to destroy the sample during the puncture test, presented in Table 3, showed two ranges of the results. The puncture work values were the highest for PA/EVOH/PE film for vacuum packaging and for OPET/PE film for MAP system, other results did not differ significantly for the rest of the tested materials. Puncture work was correlated significantly with the basic weight of films, as well as with the tensile strength and work at break at elongation test, and the breaking force at the puncture test (Table 4). In films used for food packaging in a vacuum system, the puncture work values needed to destroy the sample were higher for most materials compared to MAP films.

When measuring the extension at puncture, higher values were noted for films used in vacuum packaging than for films used for packaging in the modified atmosphere system. The results of extension at break did not differ significantly for films used for MAP packaging. The highest extension at break was obtained for PA/EVOH/PE film. Moreover, for this material the elongation was the highest at the tensile test. The results of extension at break were significantly correlated with almost all the tested physical properties and mechanical features at elongation tests of films used in the experiment (Table 4). The lowest extension was observed for PET/PA/EVOH/PE film (1.8 mm), similarly to the elongation test results which proves the low elasticity of this material.

Table 4.
Correlation matrix of the properties of tested films for vacuum and MAP packaging systems

	Basic weight	Density	Tensile modulus	Tensile strength	Elongation at tensile strength	Stress at break	Work at break	Breaking force	Puncture work	Extension at break
Thickness	0.976	ns	ns	ns	0.856	ns	0.966	ns	ns	0.731
Basic weight		ns	ns	ns	0.780	ns	0.959	ns	0.755	0.626
Density			0.908	0.651	-0.612	0.906	ns	0.905	ns	-0.687
Tensile modulus				ns	-0.743	0.854	ns	0.846	ns	-0.900
Tensile strength					ns	0.792	ns	0.864	0.901	ns
Elongation at tensile strength						ns	0.909	ns	ns	0.879
Stress at break							ns	0.853	ns	-0.638
Work at break								ns	0.606	0.713
Breaking force									0.735	ns
Puncture work										ns

ns – not significant correlation at $\alpha=0.05$

Conclusions

Based on the presented research results the following conclusions have been made:

1. The thickness and basic weight of the packaging films used in the vacuum system were higher compared with the materials for the MAP system.
2. Analyzing the results of tensile modulus and stress at break it was reported that the materials used for food packaging in the MAP system were characterized by better properties, whereas elongation at tensile strength was higher for films applied in vacuum system, except of the film with a metalized layer.
3. The highest resistance for tensile and for puncture was evaluated for OPET/PE film used in the MAP packaging. The highest elasticity showed PA/EVOH/PE film used for vacuum packaging.
4. The values of extension at puncture, breaking force and puncture work were higher for films used in packaging food with the vacuum system suggesting their better resistance for the mechanical damage by the thin pin.
5. Several correlations between the physical and mechanical properties of multilayer films have been found.

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WYBRANE WŁAŚCIWOŚCI FOLII WIELOWARSTWOWYCH STOSOWANYCH W SYSTEMACH PAKOWANIA PRÓŻNIOWEGO ORAZ W MODYFIKOWANEJ ATMOSFERZE

Streszczenie. Celem badań było określenie wybranych właściwości materiałów opakowaniowych mających zastosowanie do pakowania próżniowego i w modyfikowanej atmosferze (MAP). Badaniom poddano sześć rodzajów folii wielowarstwowych o różnym składzie, oceniając grubość, gramaturę, gęstość oraz cechy mechaniczne w testach rozciągania i przebicia. W eksperymentach wykorzystano maszynę wytrzymałościową Zwick/Roell wyposażoną w szczęki rozciągające i trzpień do przebijania. Moduł rozciągania, wytrzymałość na rozciąganie, wydłużenie przy wytrzymałości na rozciąganie, naprężenie przy zerwaniu i pracę przy zniszczeniu wyznaczono podczas testu na rozciąganie, zaś siłę przebicia, pracę przebicia i wydłużenie przy zniszczeniu oceniano za pomocą testu przebicia, przy prędkości badania 100 mm min^{-1} . Uzyskane wyniki pozwalają stwierdzić, że grubość i gramatura folii opakowaniowych stosowanych do pakowania próżniowego była wyższa w porównaniu z materiałami używanymi w systemie MAP. Analizując wyniki modułu rozciągania i naprężenia przy zerwaniu zauważono, że materiały stosowane do pakowania w systemie MAP charakteryzowały się lepszymi właściwościami, natomiast wydłużenie przy wytrzymałości na rozciąganie było wyższe dla folii używanych do pakowania próżniowego, z wyjątkiem folii z warstwą metalizowaną. Najwyższą odporność na przebicie oceniono podczas badania folii OPET/PE stosowanej w opakowaniach MAP. Wydłużenie przy zniszczeniu w teście przebicia było wyższe dla folii przeznaczonych do pakowania próżniowego, co sugeruje ich lepszą odporność na uszkodzenia mechaniczne. Stwierdzono istotne korelacje pomiędzy właściwościami fizycznymi i mechanicznymi folii wielowarstwowych.

Słowa kluczowe: polimery, folia wielowarstwowa, cechy mechaniczne, wydłużenie, przebicie

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