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COMPOSITES THEORY AND PRACTICE

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EFFECT OF SOLID STATE COMPONENTS ON THE PERFORMANCE OF MAGNETORHEOLOGICAL FLUIDS

Magnetorhelogical fluids (MRFs) are a class of multifunctional materials with the characteristics of reacting to a magnetic field by noticeable and reversible changes in their rheological and magnetic properties. The object of this study was MRFs with carbonyl iron particles based on a synthetic carrier oil. The effect of varying the mass of the solid state components such as carbonyl iron (CI) particles and fumed silica stabilizing agent additive on the performance of MRFs was examined. For this purpose, the microstructure of MRFs with different carbonyl iron addition was observed in the presence and of without a magnetic field. Moreover, the rheological properties characterization of different compositions of MRFs was conducted using a rheometer equipped with a magnetic field. It was found that the application of an appropriate proportion of solid components plays a crucial role in the formation of usable magnetorheological properties. The results of steady shear tests show that a higher mass proportion of carbonyl iron particles strongly affects the performance of MRFs, the yield stress as well as off- and on-state viscosity. A higher content of magnetic particles can ensure a substantial increase in the yield stress values of MRFs. The MRF containing 75% w/w CI achieved a yield stress at the level of 18 kPa in the magnetic field of 318 kA/m, while the MRF with lowest magnetic component mass concentration of 25% w/w reached only 0.4 kPa, whereas the MRFs with different fumed silica amounts achieved more comparable viscosity level and yield stress values. These results clearly indicate the decisive influence of carbonyl iron content on MRF performance. Oscillatory, rheometric measurements versus magnetic field show that the highest values of shear complex modulus were achieved by the MRFs with the highest percentage of carbonyl iron particles (75% w/w) and fumed silica additive (1% w/w). At the same time, the loss factor dependence on the fumed silica and carbonyl iron amount showed a much smaller effect on the MRF performance.

Keywords: **smart magnetic materials, magnetorheological fluids, rheological properties**

WPŁYW ZAWARTOŚCI FAZY STAŁEJ NA WŁAŚCIWOŚCI CIECZY MAGNETOREOLOGICZNYCH

Ciecze magnetoreologiczne należą do grupy materiałów funkcjonalnych, wykazujących w pełni odwracalną, istotną zmianę właściwości pod wpływem zewnętrznego pola magnetycznego. Przedmiot badań niniejszego opracowania stanowią ciecze magnetoreologiczne z cząstkami żelaza karbonylkowego na bazie oleju syntetycznego. Określono wpływ zawartości cząstek żelaza karbonylkowego oraz stabilizatora w postaci krzemionki koloidalnej na właściwości cieczy magnetoreologicznych. W tym celu dokonano obserwacji mikrostruktury cieczy magnetoreologicznych o różnym stopniu zawartości cząstek żelaza karbonylkowego. Badania właściwości reologicznych cieczy magnetoreologicznych o różnym składzie wykonano za pomocą reometru rotacyjnego. Wyniki badań wskazują na decydujący wpływ składu chemicznego cieczy magnetoreologicznej na kształtowanie jej właściwości użytkowych. Badania reologiczne w funkcji szybkości ścinania wykazały, że wyższa zawartości cząstek żelaza posiada silny wpływ na wartości granicy plastyczności oraz właściwości reologiczne. Większa zawartość cząstek żelaza karbonylkowego umożliwia osiągnięcie wyższych wartości granicy plastyczności. Dla porównania, ciecz magnetoreologiczna zwierająca 75% mas. żelaza osiągnęła granicę plastyczności 18kPa w polu 318 kA/m, podczas gdy ciecz z 25% mas. cząstek żelaza wykazała jedynie 0,4 kPa. Z drugiej strony, ciecze magnetoreologiczne z różną zawartością krzemionki koloidalnej charakteryzowały się zbliżonymi wartościami lepkości oraz wartościami granicy plastyczności. Efekt ten świadczy o bardziej intensywnym wpływie zawartości cząstek żelaza karbonylkowego na właściwości cieczy magnetoreologicznych. Badania oscylacyjne w funkcji pola magnetycznego wykazały, że największe wartości zespolonego modułu ścinania wykazała ciecz zawierająca najwyższą zawartość cząstek żelaza (75% mas.) oraz krzemionki koloidalnej (1% mas.). Jednocześnie wartości współczynnika stratności wykazały podobny charakter zmian i zakres wartości dla wszystkich badanych cieczy.

Słowa kluczowe: **inteligentne materiały magnetyczne, ciecze magnetoreologiczne, właściwości reologiczne**

INTRODUCTION

In recent decades, a great deal of attention has been devoted to the area of smart or multifunctional materials (SM). The key importance of SM arises from their specific capacity to receive, transmit, or process a stimulus and respond by producing a useful effect [1, 2].

One of the representatives of smart materials, with characteristics of reacting to a magnetic field with noticeable and reversible changes in their properties are magnetorheological fluids (MRFs). The considerable interest in MRFs, arising from their unique ability of rapid and reversible changes in rheological properties can clearly be evidenced by the numerous publications and patents in this area. The wide range of MRF applications comprises shock absorbers, damping devices, clutches, brakes, artificial joints and magnetorheological polishing systems [3-6].

Magnetorheological (MR) fluids are suspensions of micron-sized, magnetizable particles dispersed in a non-magnetic carrier liquid. Typical magnetic-responsive components utilized in MRFs are comprised of, e.g., carbonyl iron, iron oxide, iron alloys, nickel, cobalt, and mixtures thereof. As a carrier medium, a wide range of synthetic and mineral oils, esters, paraffins, hydrocarbons, water or other polar and non-polar liquids can be used [7]. Due to the magnetic particles/carrier fluid density mismatch, stabilizing agents are unavoidable components of MRF systems to prevent unfavourable sedimentation and agglomeration processes. For instance, thixotropic additives (e.g. fumed silica, organo-clays) and surfactants (e.g. oleic acid, aluminum stearate and lecithin) have been proven to be effective MRF stabilizators [8-12]. There are also other ways to improve MRF stability and rheological performance by adding magnetic nanoparticles or submicron-sized fillers, mixing bidisperse magnetic powders or using magnetic microfibers [13, 14].

Under an applied magnetic field, MRFs demonstrate significant and reversible changes in their rheological properties as a consequence of a dramatic transition in the microstructure. In the absence of a magnetic field, the suspension of magnetic particles is uniformly dispersed and the MRF is a free-flowing media exhibiting Newtonian behaviour. On the application of an external magnetic field, the magnetic particles become polarized and the random orientation of the magnetic particles changes to a chain-like structure, which leads to the retardation of flow (increase of apparent viscosity) and a significant change in the viscoelastic behaviour. The resulting phenomenon, called the magnetorheological effect, corresponds to a significant increase in the shear stress with a magnetically variable yield stress [7]. MRFs can exhibit a yield stress as high as 10÷100 kPa in a magnetic field of 150÷280 kA/m [5, 13]. The magnetorheological fluid performance strongly depends on many complex factors determining its applicability in a designed field. First of all, MRF compositions, i.e., magnetic particle type, dispersions and volume (mass) fraction, types and loading of stabilizing agents or type of carrier fluid determine the off- and on-state characteristics as well as intensity of the magnetorheological effect and the values of yield stress. When it comes to the external factors: strength of the magnetic field, temperature and operation mode are major parameters affecting MRF performance.

In this study, the effect of varying the mass proportion of solid components such as the magnetic particles and fumed silica additives on the performance of MR fluids with carbonyl iron is presented.

EXPERIMENTAL PROCEDURE

Carbonyl iron (CI) powder (OM, BASF) was used as the solid component for the all the examined MR fluids. This powder consists of spherical shape particles with average diameters of 5 μm (Fig. 1). Synthetic oil, polyalphaolefins (PAO), with a density of 0.9 g/cm^3 and kinematic viscosity of 100 mm^2/s at 40^{o}C, was applied as the carrier fluid. As a stabilizing agent, fumed silica (diameter 12 nm, BET 200±25) was chosen. The MRFs were prepared by mixing appropriate constituent component amounts by a mechanical stirrer until a homogeneous composition was obtained.

Three MR fluids with differing carbonyl iron particle concentrations of 75, 50 and 25% w/w, with a constant fumed silica 1% w/w additive were synthesized. With the aim of defining the stabilizer concentration effect on the rheological properties, three MRFs with the mass concentrations of fumed silica: 1, 0.5% and 0 w/w were prepared with a constant, 75% w/w of carbonyl iron powder.

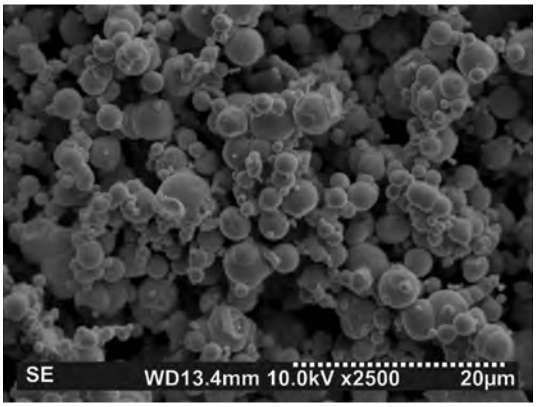

Fig. 1. SEM microphotography of carbonyl iron powder used in synthesized MR fluids

Rys. 1. Mikrostruktura SEM cząstek proszku żelaza karbonylkowego zastosowanego w wytworzonych cieczach magnetoreologicznych

The microstructural observations of MRFs with different contents of carbonyl iron powder were conducted with an optical microscope without and under a magnetic field generated by a permanent magnet.

The rheological properties of the MRFs were measured using a rotational Ares TA Instruments Rheometer, equipped with a magnetic coil. A plate-plate geometry with a diameter of 20 and 1 mm gap was used. The rheological characterization of the MRFs was carried out in the steady shear and dynamic oscillatory modes at 30^{o}C. The steady shear tests were conducted within a shear rate range of 0.1÷630 s^{-1}. The viscosity versus shear rates (viscosity curves) were obtained in the off-state (no magnetic field) and in the field

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strengths of 159 and 318 kA/m. Moreover, from the measurements of the steady shear in an external magnetic field, dynamic yield stress values for different MRFs were determined by extrapolating the shear stress-shear rate curves (not shown in this paper) to the zero shear rate.

In the oscillatory mode, the frequency of angular deformation was 6.28 rad/s (1 Hz) and the angular deformation was +/–0.5%. The measurements were carried out under a linear magnetic field increment from 0 to 239 kA/m. The complex shear storage and loss factor were determined as a function of magnetic field in order to estimate the viscoelastic properties of the MRFs.

RESULTS AND DISCUSSION

Microstructure of MRFs

In Figure 2, the microstructures of MRFs, with different carbonyl iron particles concentration, without and under an external magnetic field, are presented.

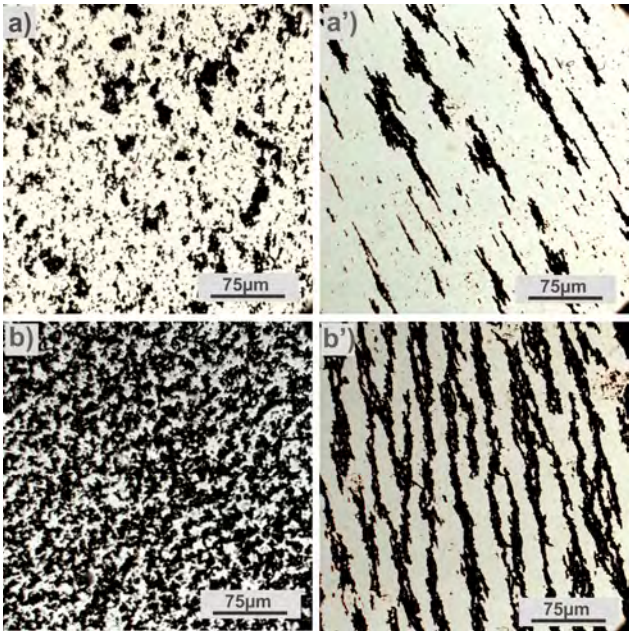

Fig. 2. Microstructure of MRFs with different concentration of carbonyl iron particles: a) 30% vol., b) 60% vol.- without magnetic field and a'), b') under applied magnetic field

Rys. 2. Mikrostruktura cieczy magnetoreologicznych z różną zawartością cząstek żelaza karbonylkowego: a) 30% obj., b) 60% obj.- brak pola magnetycznego oraz a'), b') pod wpływem pola magnetycznego

In the absence of a magnetic field, the carbonyl iron particles are uniformly distributed in the carrier oil, for both the MRFs with different iron mass concentrations. However, some aggregates of carbonyl iron particles can be observed for the more diluted MRF. Application of the magnetic field caused a dramatic transformation in the MRFs microstructure. The ferromagnetic particles became magnetically polarized and formed a chain-like structure, with a direction parallel to the magnetic field. In this case, the differences in the MRFs microstructure, with different carbonyl iron particles concentration, are more distinctive. For the more concentrated MRF, the particles formed a relatively coarse, highly networked chain-like structure, while the low concentration of carbonyl iron particles produced a sparse chain formation. Therefore, for enhanced magnetorheological performance of an MRF, a highly-networked chain structure would be more favourable.

The effect of carbonyl iron powder content on rheological performance of MRFs

The viscosity curves for MRFs with different carbonyl iron particles mass content, without a magnetic field and in 159 and 318 kA/m are presented in the Figure 3. As can be seen, the MRF with 25% w/w magnetic particles shows the lowest off-state viscosity. The off-state viscosity of fluids is an important factor, and generally the lower the off-state viscosity, the larger range of achievable viscosity may be adjusted. For the MRF with 75% w/w carbonyl iron particles, the off-state viscosity reaches the highest value of 315 Pa·s. All the investigated MRFs display a viscosity decrease with an increasing shear rate, evidencing non-Newtonian behaviour, defined as shear thinning.

Application of the magnetic field causes a significant change in the viscosity values for all the examined MRFs. In the magnetic field of 318 kA/m, the MRF with 75% w/w CI exhibits a maximum viscosity of 188.8 $\cdot 10^3$ Pa·s. Simultaneously, the MRFs with 50 and 25% w/w CI exhibit 47.9 and $5.7 \cdot 10^3$ Pa·s, respectively.

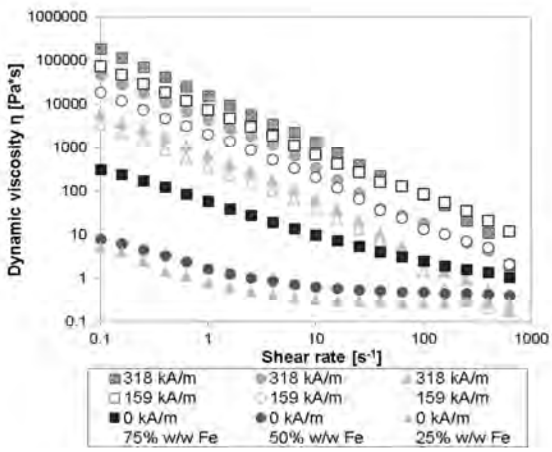

Fig. 3. Dynamic viscosity as function of shear rate for MRFs with different carbonyl iron particles amount, without magnetic field and in 159 and 318 kA/m

Rys. 3. Lepkość dynamiczna w funkcji szybkości ścinania dla cieczy magnetoreologicznych z różną zawartością żelaza karbonylkowego, w polach 159 i 318 kA/m

Obviously, the values of the MRFs on-state viscosity for a given magnetic field increases with an increase in the magnetic particles mass fraction. These results are directly related to the field induced structure depending on the load of magnetic particles. In the case of the MRFs with the high carbonyl iron particle mass concentration, the chain-like formation of magnetically polarized particles is highly networked and thus, the flow resistance of such a suspension is restricted, which consequently leads to higher viscosity values.

In Table 1, the yield stress values for the MRFs with different carbonyl iron % w/w concentration in a magnetic field of 159 and 318 kA/m are compared. The yield stress in the MRFs can be described as the critical stress required to destroy the chain-like structure in a magnetic field.

TABLE 1. **Yield stress for MRFs with different carbonyl iron particle amounts**

TABELA 1. **Wartości granicy plastyczności dla cieczy magnetoreologicznych z różną zawartością cząstek żelaza karbonylkowego**

Sample MRF No.	Carbonyl iron [% w/w]	Yield stress in 159 kA/m, τ_{159} [kPa]	Yield stress in 318 kA/m, τ_{318} [kPa]
1	75	7.56	18.87
2	50	2.28	4.78
3	25	0.42	0.69

It can be noticed that a higher content of magnetic particles can ensure a substantial increase in the yield stress values. The MRF with 75% w/w CI achieves a yield stress at the level of 18 kPa in the magnetic field of 318 kA/m, while the MRF with the lowest magnetic component concentration of 25% w/w reaches only 0.4 kPa. The effect of the magnetic particles mass concentration on the yield stress can be correlated with the field induced structure, which enhances the resistance of MRFs to flow. It is evidenced that a higher magnetic particles concentration can create stronger field-induced forces and in consequence, higher values of yield stress.

The viscoelastic parameters such as complex shear modulus G*, and loss factor tan(δ), for the MRFs with various carbonyl iron mass concentrations, versus a magnetic field of 0÷230 kA/m, are shown in Figure 4.

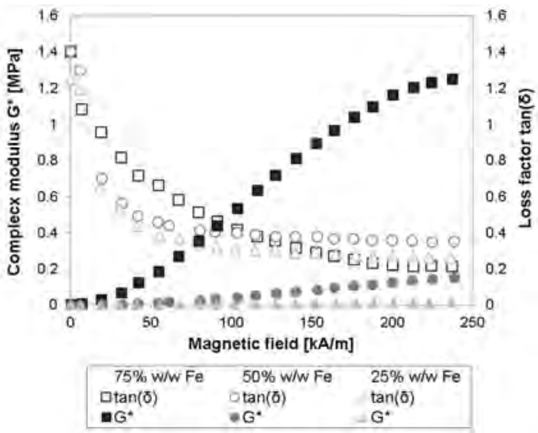

Fig. 4. Dependence of complex modulus and loss factor on an applied magnetic field for 75, 50 and 25% w/w carbonyl iron MRFs

Rys. 4. Zależność modułu zespolonego i współczynnika stratności od pola magnetycznego dla cieczy magnetoreologicznych z masową zawartością żelaza 75, 50 oraz 25% masowych

Storage modulus G' is a measure of the energy stored elastically during deformation, while loss modulus G" quantifies the energy dissipated into heat. Complex modulus G* refers to the overall viscoelastic properties and quantifies the total resistance to the oscillatory flow. These parameters can be applicable in predicting field-induced aggregation of the MRF under the external magnetic field.

The loss factor is the ratio of loss modulus to the storage modulus and thus provides a measure of the relative viscous and elastic behaviour.

In the current experiments, all the three MRFs reveal a similar behaviour of shear complex modulus and loss factor. Application of the magnetic field results in a substantial increase (three orders of magnitude) in the complex shear modulus due to the induced interparticle forces. At the same time, the loss factor values decrease with an increasing magnetic field strength indicating the dominating elastic behaviour in the overall viscoelastic character of the MRFs. The intensity of change in the shear complex modulus and loss factor depends on the amount of carbonyl iron particles. The highest complex shear modulus G* of 1.25 MPa was exhibited by the fluid with the highest carbonyl iron loading (75% w/w) in a magnetic field of 239 kA/m. Noticeably, the MRF with 25% w/w carbonyl iron particles reaches a considerably lower value of shear modulus G* = 0.15 MPa at the same magnetic field. This result clearly indicates that the carbonyl iron content affects the field induced modulus. A higher weight fraction of magnetic particles provides stronger magnetic induced interactions between the particles and leads to a substantial increase in the complex shear modules. Comparing the loss factor for the MRFs with various amounts of carbonyl iron particles, it can be observed that they are very similar. Moreover, all the examined suspensions demonstrate a transition from viscous into elastic behaviour at the same magnetic field of 7 kA/m.

The effect of silica fumed stabilizer content on rheological performance of MRFs

The fumed silica component in the MR suspensions constitutes a thixotropic agent with the role of stabilizing the performance of MRFs and counteracting the sedimentation process of the magnetic particles.

In the MRFs, fumed silica forms a thixotropic network stable at low shear rates, but easily disrupted when a stronger shearing force is applied.

The effect of the varied fumed silica stabilizing agent content on the viscosity of MRFs is presented in Figure 5.

The MRF with the higher fumed silica percentage presents a higher off-state viscosity versus shear rate, especially at lower shear rates. Obviously, the lowest off-state viscosity of 17 Pa·s is exhibited by the MRF without the addition of fumed silica. Low viscosity is a convenient parameter, however, the lack of a stabilizer may cause agglomeration of the particles, which in

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the presence of the magnetic field may not align in separate chains but form larger aggregates, leading to deterioration of the rheological properties. Therefore, using stabilizing agents is an indispensable way to produce a stable dispersion of magnetic particles, even though the off-state viscosity is increased.

Clearly, the highest viscosity values (315 Pa·s) are obtained by the MRF with the highest amount of fumed silica.

As can be noticed, all the three MRFs, with various fumed silica content exhibit comparable on-state viscosity at the level ranging from $1.89 \cdot 10^5$ to $1.58 \cdot 10^5$ Pa·s, at the maximum applied magnetic field (318 kA/m), at the shear rate of 0.1 s^{-1}. Additionally, the viscosity values decrease intensely with increasing shear rates for all the examined fluids, which indicates shear thinning characteristics.

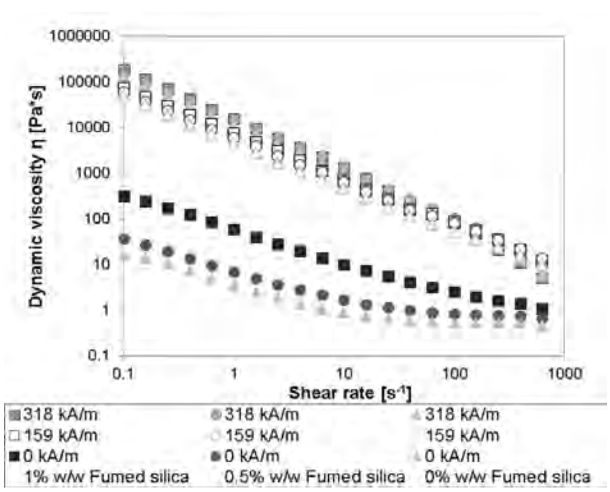

Fig. 5. Dynamic viscosity as a function of shear rate for MRFs with different fumed silica amounts, without magnetic field and in 159 and 318 kA/m

Rys. 5. Lepkość dynamiczna w funkcji szybkości ścinania dla cieczy magnetoreologicznych z różną zawartością krzemionki koloidalnej, bez pola i w polu magnetycznym 159 i 318 kA/m

The values of yield stress for the MRFs with different loading of fumed silica are collected in Table 2.

TABLE 2. **Yield stress for MRFs with 75% w/w carbonyl iron and different fumed silica amount**

TABELA 2. **Wartości granicy plastyczności dla cieczy magnetoreologicznych z zawartością 75% żelaza i różną zawartością krzemionki koloidalnej**

Sample MRF No.	Fumed Silica [%w/w]	Yield stress in 159 kA/m, τ_{159} [kPa]	Yield stress in 318 kA/m, τ_{318} [kPa]
1	0	5.07	13.11
2	0.5	6.06	15.82
3	1	7.56	18.87

As can be observed, in the magnetic field of 159 kA/m all three MRFs exhibit comparable yield stress values with the tendency of higher values for an increasing fumed silica amount. In the magnetic field of 318 kA/m, the highest yield stress is achieved by the MRF with 1% w/w fumed silica. This effect may be contributed to better distribution of the magnetic particles in the case of the MRFs with a higher fumed silica additive, due to the formation of a thixotropic network by the silica particles.

The viscoelastic parameters versus the magnetic field for the MRFs differing in fumed silica amount are presented in Figure 6.

Increasing the fumed silica mass concentration in the MRFs results in higher values of shear module. The MRFs without and with 0.5% w/w silica additive exhibit a comparable complex modulus of 0.7÷0.8 MPa at the highest applied magnetic field (239 kA/m). The fumed silica additive of 1% w/w causes a substantial increase in the maximum value of the MRF shear modulus up to 1.2 MPa at 239 kA/m. All the three MRFs exhibit comparable values of loss factor which indicates a similar viscoelastic behaviour. The transition point, correlating to the value of magnetic field when the loss factor achieves a value below 1 and the elastic behaviour prevails over the viscous response, is similar for the three examined fluids (> 7 kA/m). Under a magnetic field of 7 kA/m, the loss factor reveals domination of the viscous properties by the elastic behaviour and is associated with the field-induced structure.

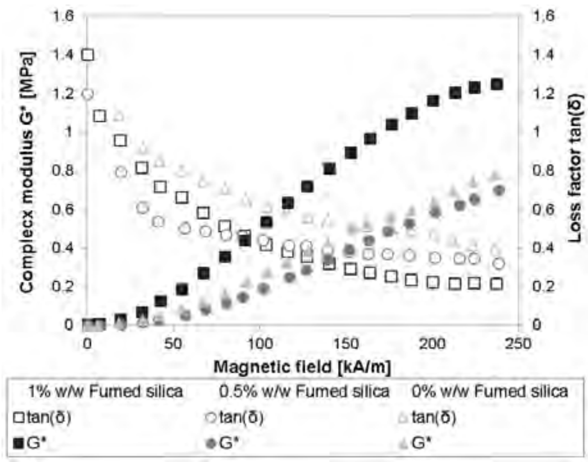

Fig. 6. Dependence of complex modulus and loss factor on applied magnetic field for MRFs with different percentage of fumed silica addition

Rys. 6. Zależność modułu zespolonego i współczynnika stratności w funkcji pola magnetycznego dla cieczy magnetoreologicznych z różną zawartością krzemionki koloidalnej

CONCLUSIONS

A series of magnetorheological fluids was synthesized on a basis of synthetic oil with various contents of carbonyl iron particles and fumed silica stabilizing agent.

It was found that the application of an appropriate composition of solid state components plays a crucial role in the formation of usable magnetorheological properties.

An appropriate carbonyl iron particle content affects the performance of MRFs as the magnetic field induced structure can form more networked chains, giving rise to a substantial change in the rheological properties. The results of steady shear tests show that a higher carbonyl iron particles fraction strongly affects the yield stress as well as the off- and on-state viscosity. A higher content of magnetic particles can ensure a substantial increase in the yield stress values of MRFs. The MRF with 75% w/w CI achieves a yield stress at the level of 18 kPa, in a magnetic field of 318 kA/m, while the MRF with the lowest magnetic component concentration of 25% w/w reaches only 0.4 kPa. In contrast, the MRFs with different fumed silica amounts exhibit more comparable viscosity and yield stress values. These results clearly indicate the prevailing influence of carbonyl iron content on MRF performance.

Oscillatory measurements in the function of magnetic field show that the highest values of shear complex modulus were achieved for the MRF with the highest content of carbonyl iron particles (75% w/w) and fumed silica additive (1% w/w). At the same time, the loss factor is much less sensitive to fumed silica and carbonyl iron concentration.

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