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INFLUENCE OF HEAT TREATMENT ON SUSCEPTIBILITY TO HOT CRACKING OF MAGNESIUM ALLOY EN-MCMgRE3Zn2Zr

WPŁYW OBRÓBKI CIEPLNEJ NA SKŁONNOŚĆ DO PĘKANIA GORĄCEGO STOPU MAGNEZU EN-MCMgRE3Zn2Zr

Alloy EN-MCMgRE3Zn2Zr is gravity casted to sand casting moulds and is used mainly in aviation industry and car industry. In castings of magnesium alloy inconsistencies in form of porosities or misruns often appear. Such defects are repaired with the use of overlay welding and welding techniques. Main difficulty during welding magnesium alloys is their susceptibility to cracking in crystallization. These cracks are results of effects proceeded in Brittleness Temperature Range (BTR). It is temperature range between liquidus temperature and loss ductility temperature i.e. temperature on heating at which the ductility of material drops to zero.

Technological factors, which decide above BTR are: welding condition and structure of cast formed in casting and heat treatment. Metallurgical factors are: chemical composition of alloys, chemical reaction during welding and morphology of joint. Constructional factors, such as shape of casting mould, his specific stiffness (quotient Young's modulus and density) decide about size of stresses and deformation accompanying formation of joint weld.

In this paper an assessment is presented of the influence of technological, metallurgical and constructional factors and, in particular, the influence of heat treatment on the size and character of high-temperature brittleness range of the casted magnesium alloy EN-MCMgRE3Zn2Zr. The range of research included overlay welding and welding tests, metallographic research and the assessment of the mechanical properties in crystallisation temperature range. It was stated that EN-MCMgRE3Zn2Zr alloy is in a group of well weldable alloys with the use of tungsten inert gas welding, independently of the conducted heat treatment. A repair technology of the gravity castings made of EN-MCMgRE3Zn2Zr was worked out.

Keywords: Weldability of the magnesium alloys; EN-MCMgRE3Zn2Zr; temperature brittleness range, hot cracking, repair of castings

Stop magnezu EN-MCMgRE3Zn2Zr jest odlewany grawitacyjnie do form piaskowych. Stosowany jest najczęściej w przemyśle lotniczym i samochodowym. Po procesie odlewania w materiale mogą pojawić się niedolania i rzadzizny. Wady te naprawiane są z zastosowaniem technik napawania i spawania.

Główną trudnością praktyczną przy spawaniu i napawaniu stopów magnezu jest ich skłonność do pękania gorącego w procesie krystalizacji. Pęknięcia te są wynikiem zjawisk zachodzących w tzw. zakresie kruchości wysokotemperaturowej (BTR), rozumianym jako zakres temperatur pomiędzy temperaturą likwidus a temperaturą utraty plastyczności, tj temperaturą podczas nagrzewania, w której plastyczność materiału zmierza do zera.

Czynnikami technologicznymi decydującymi o BTR są warunki spawania określające ilości wprowadzonego ciepła, a zarazem decydujące o szybkości chłodzenia odlewu oraz struktura odlewu kształtowana w procesie odlewania i obróbki cieplnej odlewów. Do czynników metalurgicznych wpływających na BTR zaliczyć należy: skład chemicznych stopu, reakcje chemiczne zachodzące podczas spawania oraz morfologię struktury złącza. Na wielkość naprężeń i odkształceń towarzyszących powstawaniu złącza mają wpływ czynniki konstrukcyjne m.in. forma odlewu i jego sztywność właściwa rozumiana jako iloraz modułu Younga i gęstości (E/ρ).

W pracy przedstawiono ocenę wpływu czynników technologicznych, metalurgicznych i konstrukcyjnych na wielkość i charakter zakresu kruchości wysokotemperaturowej odlewniczych stopów magnezu. Badania obejmowały próby napawania i spawania, badania metalograficzne oraz wyznaczenie temperaturowego zakresu kruchości.

1. Introduction

Magnesium alloys together with aluminium alloys and titanium alloys belong to light alloys group, which has the biggest practical importance in applications in constructions.

Four main directions of magnesium alloys development can be enumerated [1-3]. They are all connected

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with the decrease in weight of elements with preservation or improvement of their previous properties (Fig. 1). First two directions are aimed at achievement of wrought alloys and gravity casted alloys, characterised with good plasticity and big strength. Among the most important here, there are modified alloys Mg-Al and Mg-Si and alloys with addition of lithium. Third direction is the group of composites on the matrix of magnesium alloys showing good creep resistance and high Young's modulus. The biggest number of tests, however, is conducted in order to increase the creep resistance which will enable the application in higher exploitation temperatures (Fig. 1).

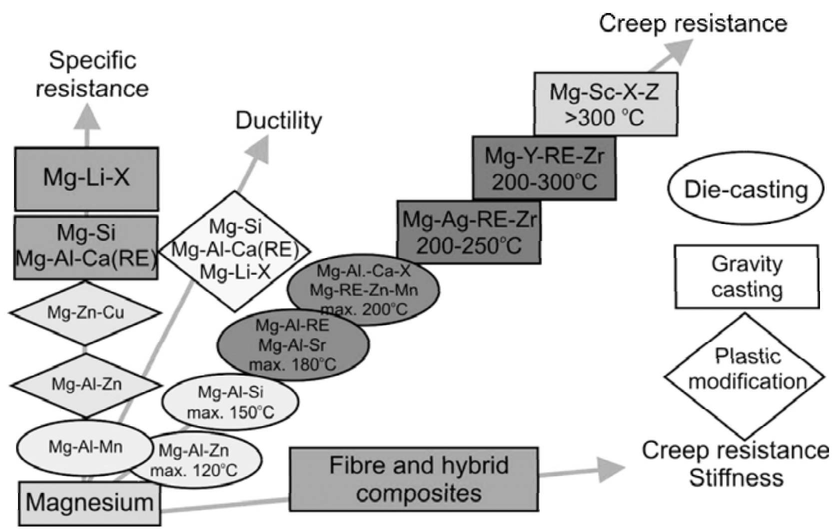

Fig. 1. Development directions of magnesium alloys [2]

Magnesium alloys are presently used for casting of huge-dimensions casts to sand moulds, high-pressure casts and precise casts [3]. Defects and casting inconsistencies often appear in castings from magnesium alloys (misruns, porosities and cracks), especially in huge-dimensions casts. The frequency of the appearances of casting defects for castings with welding techniques. It a practice commonly used in foundries. Welding techniques can also be applied in case of joining elements made of Mg alloys and for regeneration of castings after their service consumption. Number of castings of magnesium alloys which are repaired and constructions which are welded is not satisfactory and it often makes the production unprofitable [4].

Alloys from group Mg-Zn-RE-Zr can be divided into two groups: with increased content of zinc (EN-MCMgRE3Zn2Zr, ZE41, ZE63) and with increased content of rare earth elements (MEZ) [2,5]. The structure of alloys Mg-Zn-RE-Zr with increased content of rare earth elements is characterised with the presence of solid solution grains α-Mg and inter-metallic phase divisions $Mg_{12}RE$. Because of higher zinc content, in EN-MCMgRE3Zn2Zr alloy on grain boundaries of solid solution α-Mg a phase $(Mg, Zn)_{12}RE$ forms, which is a secondary solid solution on the matrix of inter-metallic phase $Mg_{12}Ce$ [5]. Such alloys are applied in aviation and car industries for complex castings which work in creep conditions.

Weldability can be defined as the ability of the material to create joints with the use of welding, which meet the requirements set for them [4]. In literature it is most often suggested to use tungsten inert gas welding (TIG) for welding magnesium alloys, because of its precise regulation of the applied heat amount. However, independently from the applied method, by welding magnesium alloys some difficulties appear, connected mainly with their metallurgical properties such as [1,2,4]: chemical composition, big affinity of Mg to oxygen and formulation of MgO oxide with a very high melting temperature of about 2500°C, high heat conduction of Mg and its alloys, low evaporation temperature of the alloys, about 1100°C, brittleness in temperature range of about 400°C, wide temperature range between the liquidus and the solidus, deciding about the so-called high-temperature brittleness range (HTBR) and the amount and arrangement of segregating eutectic, the so-called "residue liquid".

The most frequent reason for disqualification of welded joints from casted magnesium alloys are the hot cracks. The decrease in metal ductility in high-temperature brittleness range and metal deformation during crystallisation, when, as a result, the material resistance loss occurs; are the main reasons of hot cracks formulation. Upper limit of this range during welding is the nil-strength temperature NST and the lower limit is assumed as the nil-ductility temperature NDT during heating or ductility recovery temperature DRT during cooling [1].

Technological factors deciding upon weldability of the magnesium alloys are: type of joint, filler metal and the method and conditions of welding which decide about the amount of the introduced heat (arc linear energy, kJ/cm) and at the same time about the cooling speed of weld and the heat affected zone.

According to the producer, the ZRE-1 alloy can be welded with the use of TIG method, with the use of filler metal with the same chemical composition [2]. There are, however, no tests conducted by independent units, which would confirm such information.

2. Research material

A magnesium alloy with the addition of zinc and rare earth elements called EN-MCMgRE3Zn2Zr was used in tests. To assess the influence of metallurgical, technological and constructional factors the alloys were used both in delivery state, which means after casting to sand moulds in the form of pig sows and after heat treatment. Chemical composition, alloy properties and heat treatment parameters are presented in table 1.

TABLE 1

Chemical composition and properties of alloy EN-MCMgRE3Zn2Zr. EN-MC MgRE3Zn2Zr

	Chemical composition, %				
EN-MC MgRE3Zn2Zr	Melt	Zn	Zr	RE	Other
	BS EN 1753	2.0-3.0	0.4-1.0	2.5-4.0	–
	20091901	2.8	0.51	2.87	<0.05
Mechanical properties					
EN-MCMgRE3Zn2Zr		R_m, MPa	R_e, MPa	A_5,%	HV3
		160	110	3	50
Heat treatment		Alternative I	Stress relief annealing: 10h/180°C/air		
		Alternative II	Stress relief annealing: 16h/200°C/air		

3. Overlay welding and welding tests

Overlay welding and welding tests were conducted on 5mm thick plates cut out from gravity casts. The cut out samples were heat treated with the use of two treatment alternatives (table 2). In case of first alternative, the samples were heated to a temperature of 180°C, kept for 10 hours and then cooled in air. Second alternative of heat treatment consisted of heating the samples to 200°C, holding them for 16 hours and then cooling in air. Filler metal with chemical composition close to basic material was used in welding: 2.5 % Zn, 0.52 % Zr i 3.17 % RE in the form of a wire with diameter of 2.4mm.

Welding and overlay welding was conducted with the use of tungsten inert gas welding (Ar 99,995%) (TIG) with inverter source Invertec 240 AC/DC. Weld penetrations without filler metal were conducted to determine the basic parameters of the process. Next, grooves were made on samples to simulate the casting defects and they were padded with one or more beads. Optimisation of welding parameters enables the choice of parameters to perform a butt weld. Technological parameters of overlay welding and welding are presented in table 3 and the example faces of weld penetrations are shown in figure 4.

Visual tests conducted according to EN ISO 970 standard requirements have shown the uneven face of weld penetration conducted without additional wire. In case of weld penetration with additional wire, the faces of overlay welds were correct and characterised with smoothness (Fig. 2). In one-layer overlay weld, padded with current of 140A, in material in delivery state, a hot crack in the weld axis was found (Fig. 2b). The tests of two-sided butt welds did not show the welding inconsistencies, either on the side of weld face or root of weld.

TABLE 2

Presentation of overlay welding and welding parameters of alloy EN-MCMgRE3Zn2Zr

Type of joint	Number of runs	Additional material	Current intensity, A	Current voltage, V	Gas Ar, l/min	Arc linear energy, kJ/cm
overlay weld	1	No	80	15	14	2.4
overlay weld	1	No	100	15	14	3.0
overlay weld	1	No	120	15	14	3.6
overlay weld	1	Yes	140	15	14	4.2
overlay weld	2	Yes	130	15	14	3.9
overlay weld	2	Yes	140	15	14	4.2
weld	3	Yes	130	15	14	3.9

4. Macro- and micro-structure analysis

Samples for tests were cut out perpendicularly to welding direction, next they were grinded and polished on diamond pastes according to regulations of the procedure worked out in Faculty of Materials Science in Silesian University of Technology [3]. The micro-sections prepared in such a way were etched in reagent: 19 ml of water, 60 ml of ethylene glycol, 20 ml of acetic acid, 1 ml of HNO_3. Macroscopic observations were conducted on stethoscope microscope Olympus SZX9 with magnifications of 20 xs in dark field technique. The aim of research was to determine the geometric sizes of the fusion and to show the welding inconsistencies. Examples of padded welds and a weld for material in delivery state are presented in Fig. 3.

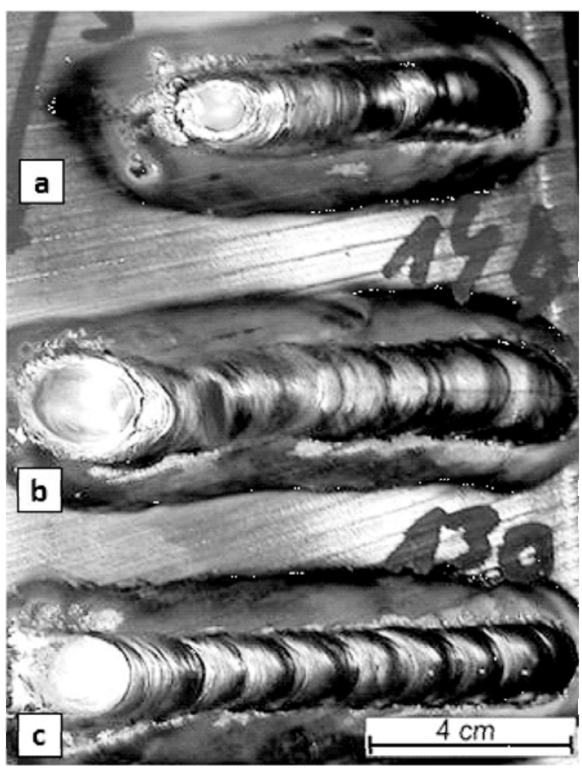

Fig. 2. Faces of weld penetration for EN-MCMgRE3Zn2Zr alloy in delivery state conducted with additional wire in TIG method, welding current: a) 120A, b) 140A, c) 130A

Microstructure tests were conducted on light microscope Olympus GX-71 in light field technique, with magnifications from 50 to 500xs. The found microstructures after heat treatment for butt joints are shown in Fig. 4a, b, whereas the path of crack in weld axis in material in delivery state is presented in Fig. 4c.

In tested padded welds and welds a correct shape and the right joining with the parent material were found

(Fig. 3). No micro-cracks or other welding inconsistencies in the welded joints after heat treatment were found, whereas the crack in the weld penetration axis, performed in the material in delivery state, was confirmed. The crack propagates throughout the face of padded weld through the heat-affected zone to the base material (Fig. 4c).

Conducted tests confirmed the right choice of overlay welding and welding technologies, which is shown by the right construction of the joint and, in particular, the heat-affected zone and weld penetration area. The geometric coefficient of the joint, calculated as the ratio of width to the height of weld penetration on flat perpendicular cross-section was on a level of 4 for padded welds and around 1 for butt welds. No significant influence of the heat treatment was confirmed on the geometrical shape of the weld penetrations.

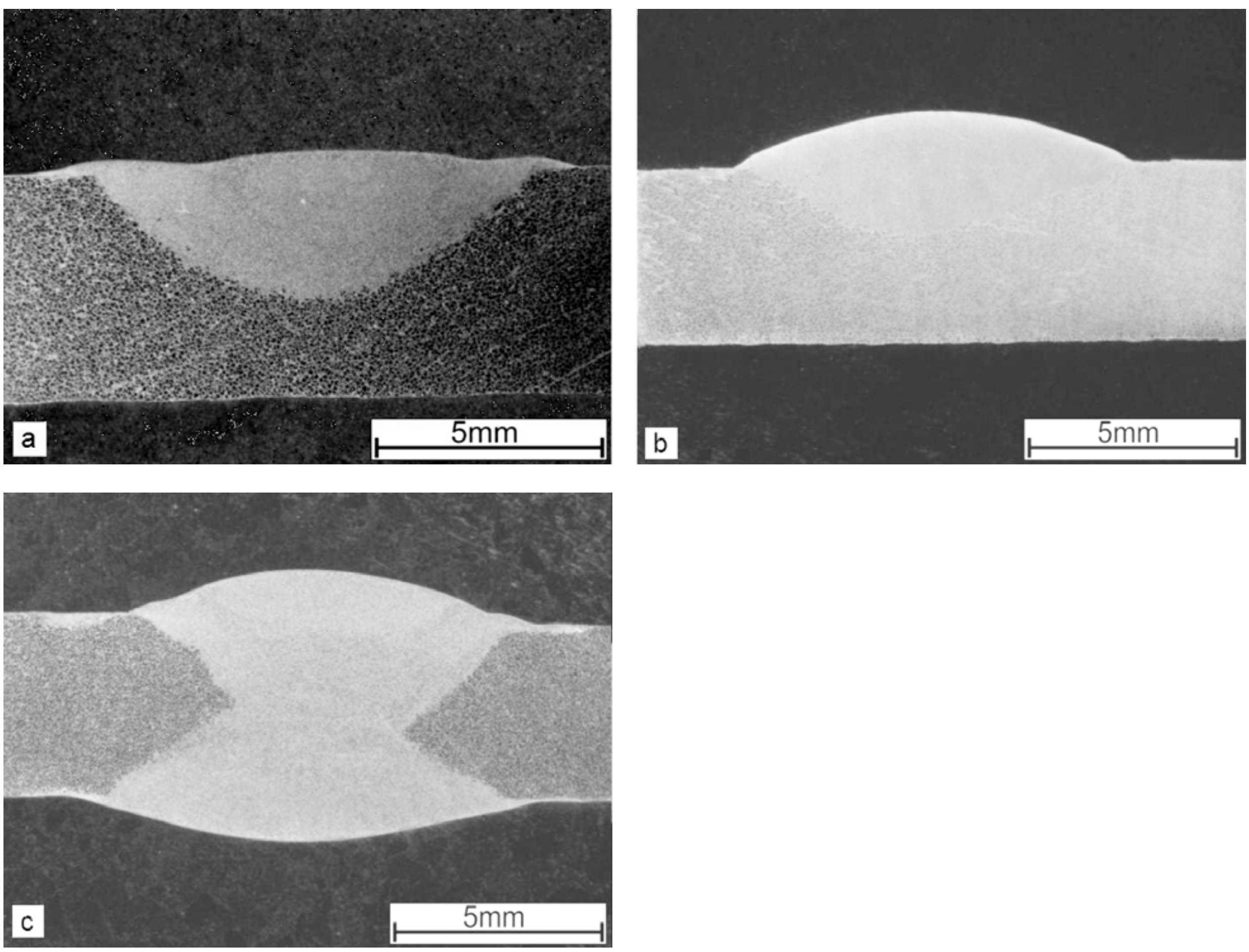

Fig. 3. Macrostructure of the conducted padded welds and butt joints of EN-MCMgRE3Zn2Zr alloy in delivery state

Tests of structure have shown the typical areas of the welded joint, which are: the parent material, the fusion penetration area including fusion line and the heat-affected zone (Fig. 4a). Tests of the found crack show that the crack runs along the crystal boundaries of solid solution Mg (α), through the disruption of the partially melted liquid film in the area of inter-metallic phases (Fig. 4c).

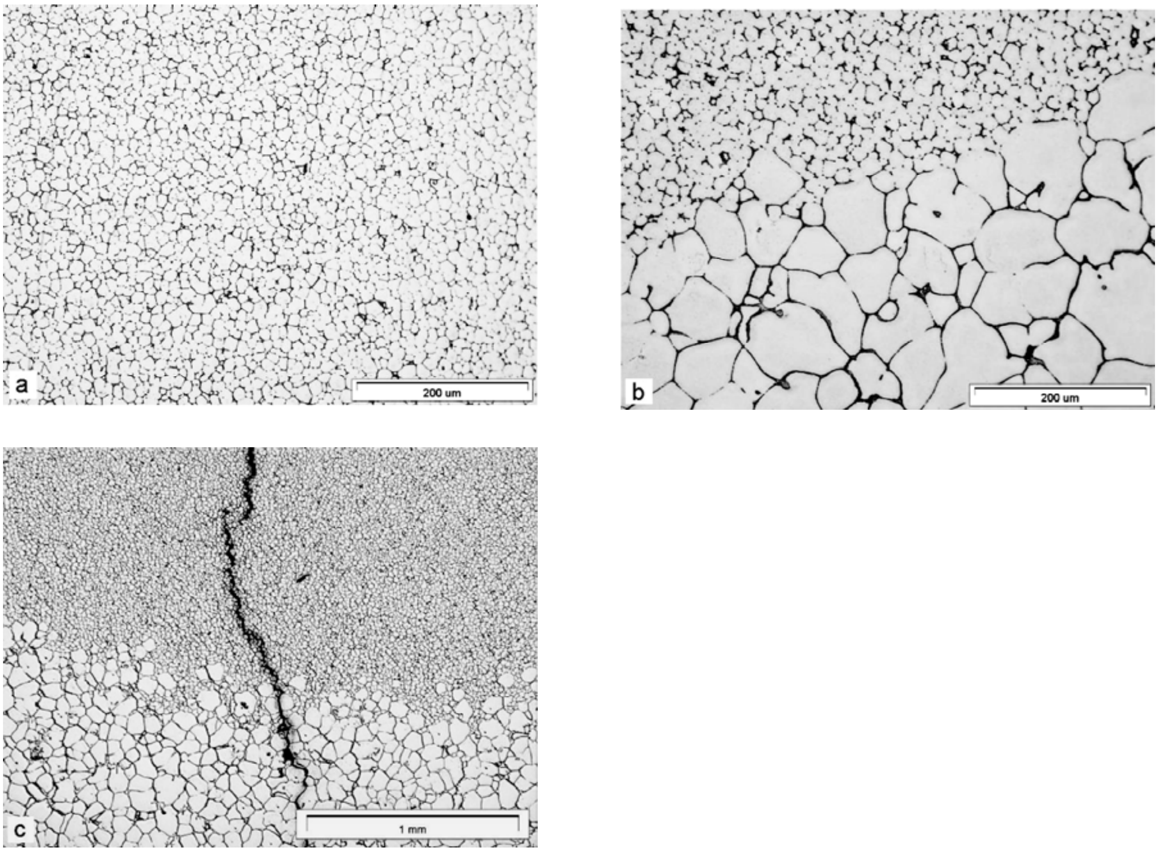

Fig. 4. Microstructure of the welded joint of EN-MCMgRE3Zn2Zr alloy after heat treatment of basic material: a) area of padded weld, b) fusion line of padded weld, c) crack in the padded weld axis of the material in delivery state, welding current 140A

5. Hardness tests

Hardness tests of the joints of alloy EN-MCMgRE3Zn2Zr in delivery state and after heat treatment were the complement of the structural tests. The measurements were conducted on automatic hardness testing machine Abramin A-300 by Struers company, with the use of Vickers HV3 method. Achieved results are graphically presented in fig. 5.

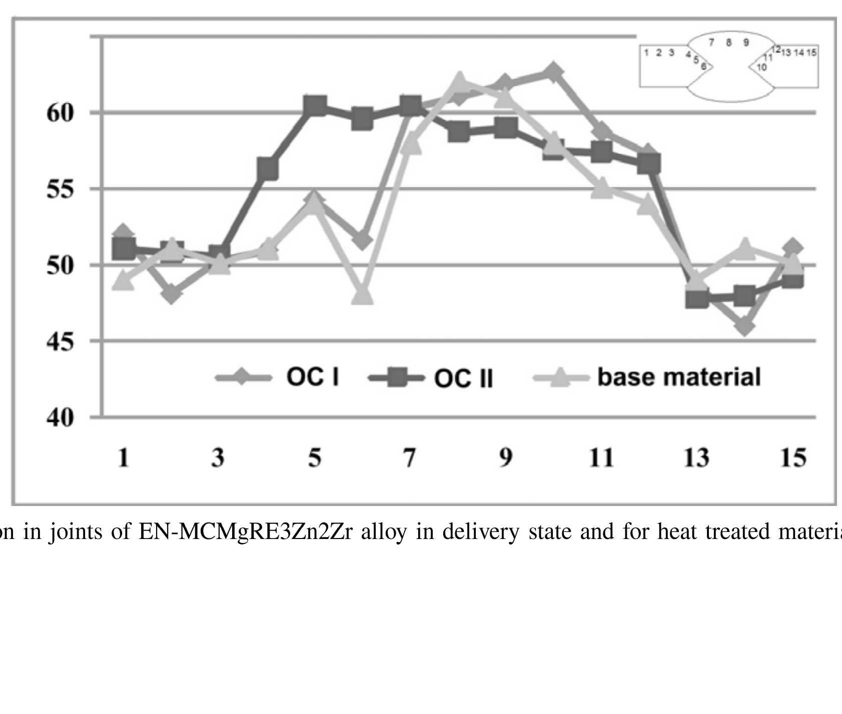

Fig. 5. Hardness distribution in joints of EN-MCMgRE3Zn2Zr alloy in delivery state and for heat treated material before welding (table 2)

6. Determination of temperatures characteristic for crystallisation and melting process of alloy EN-MCMgRE3Zn2Zr

Temperatures characteristic for crystallisation and melting process of EN-MCMgRE3Zn2Zr alloy were determined with the use of differential thermal analysis DTA. Tests were conducted on thermal analyser SETSYS by Setaram company, with the use of TG-DTA head. The system enables to measure the heat flow in phase changes connected with melting and crystallisation of the tested alloys. The measurement of the beginning and end of the change was conducted with the use of one set point method. Conditions of experiment and marked temperatures are presented in table 3.

TABLE 3

Heating and cooling conditions during differential thermal analysis for tested magnesium alloys

Name of alloy	Heating temperature, °C	Heating speed, /cooling speed, °C/min	Atmosphere, %	Gas flow speed, l/h	Type of furnace thermoelement
MCMgRE3Zn2Zr	750	10	Ar 99,999	1.45	type S (Pt-Rh 10%)
heating		Temperature of melting beginning of inter-metallic phase, °C	Temperature of melting end of inter-metallic phase, °C	Temperature of melting beginning of solid solution Mg(α), °C	Liquidus temperature, °C
		570	616	616	680
cooling		Temperature of crystallisation beginning of solid solution Mg(α), °C	Temperature of crystallisation end of solid solution Mg(α), °C	Temperature of crystallisation beginning of inter-metallic phase Mg(α), °C	Solidus temperature, °C
		644	598	572	552

7. Determination of Nil-strength temperature (NST)

Tests on cylindrical samples size Ø 6 x 90mm on Gleeble 3800 simulator were conducted in order to determine the nil-strength temperature (NST) during heating. Thermoelements type S were bonded to the samples and next fixed with the use of copper fixtures in the chamber of the device. A constant distance of the fixtures of 52.4mm was maintained. The chamber, after removal of air, was filled with argon (to 0.14hPa). Next, a minimum initial load was applied of 0.6 – 0.7kN, which was maintained until the end of experiment. Samples were heated with the speed of 20°C/s to a temperature of 400°C, and then with a speed of 1°C/s. The temperature, in which the cracks appeared, was defined as NST temperature. It was stated in all of the cases, independently from the heat treatment, that the NST temperature is 535°C. Microstructure of the crack area discovered on microsection perpendicular to fracture surface and the fracture surface of the tested alloy is presented in fig. 6.

8. Determination of Nil-ductility temperature (NDT), ductility recovery temperature (DRT) and high-temperature brittleness range (HTBR)(in Polish – ZKW)

In order to assess the width of high-temperature brittleness range HTBR and to determine the influence of the structure on hot cracking mechanism the nil-ductility temperature NDT during heating and the ductility recovery temperature DRT during cooling were determined. The temperature, in which the contraction of sample is below 3% was assumed as NDT, and temperature of above 3% contraction was assumed as DRT.

Tests were conducted on Gleeble 3800 device. Cylindrical samples size Ø 10 x 120mm were placed in protective argon atmosphere and fixed in copper fixtures. To determine the NDT the samples were heated to a given temperature in high-temperature brittleness range, but below the NST temperature and soaked for 5s, and next stretched with marked, constant speed. Ductility recovery temperature DRT was marked during cooling of samples to temperature close to NST to given temperature value and next deforming the sample with constant speed of 20 mm/s.

Marked NDT and DRT temperatures, mechanical properties of EN-MCMgRE3Zn2Zr alloy in those temperatures, value of coefficient R_f and the calculations results of HTBR width are presented in table 5. Achieved results enabled to determine the dependence of ductility understood as contraction to the resistance in temperature function during heating and cooling of the tested alloy (fig. 7).

TABLE 4

Parameters and results of NDT and DRT tests for alloy EN-MCMgRE3Zn2Zr

Alloy	OC	V_o, mm/s	Δl, mm	Z, %	R_m, MPa	NDT, °C	R_f	
NDT	–	20	0,6	2,9	53	500	0.37	
	I	20	1,0	2,1	49	500	0.37	
	II	20	1,0	1,8	59	495	0.37	
Alloy	OC	V_o, mm/s	Δl, mm	Z, %	R_m, MPa	DRT, °C	ZKW, °C	ΔZKW, °C
DRT	–	20	1,0	2,3	51	490	535-490	45
	I	20	0,3	1,9	50	500	535-500	35
	II	20	0,8	1,9	59	500	535-500	35

where: OC - heat treatment according to table 2., V_o – deformation speed, NDT- nil-ductility temperature, R_f – coefficient of resistance to hot cracking, DRT – ductility recovery temperature, Polish- ZKW-English-HTBR – high-temperature brittleness range, ΔZKW – width of high-temperature brittleness range

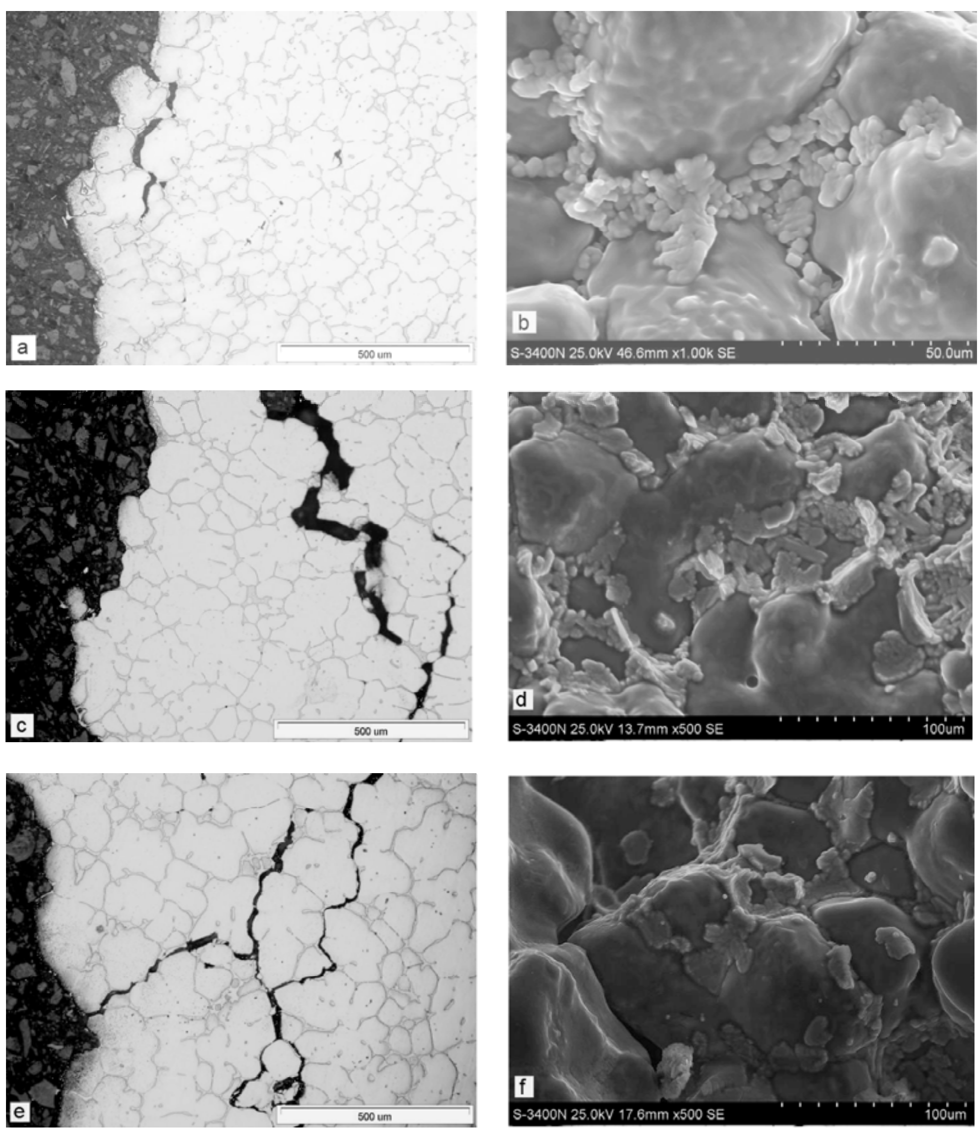

Fig. 6. Structure of the crack area of EN-MCMgRE3Zn2Zr alloy after NST test: a)microstructure of the area perpendicular to the fracture of alloy in initial state with visible subsurface crack, LM, b) phase divisions $(Mg,Zn)_{12}RE$ on surface of alloy fracture in initial state, SE picture, c) post-fracture crack along the crystals boundaries Mg(α) in alloy after stress relief annealing (OC I), LM, d) topography of the alloy fracture after stress relief annealing with phase divisions $(Mg,Zn)_{12}RE$ on crystals boundaries Mg(α)(OC I), SE, e) network of cracks with visible bridges between crystals, (OC II), LM, f) phase divisions $(Mg,Zn)_{12}RE$ and inter-crystalline crack

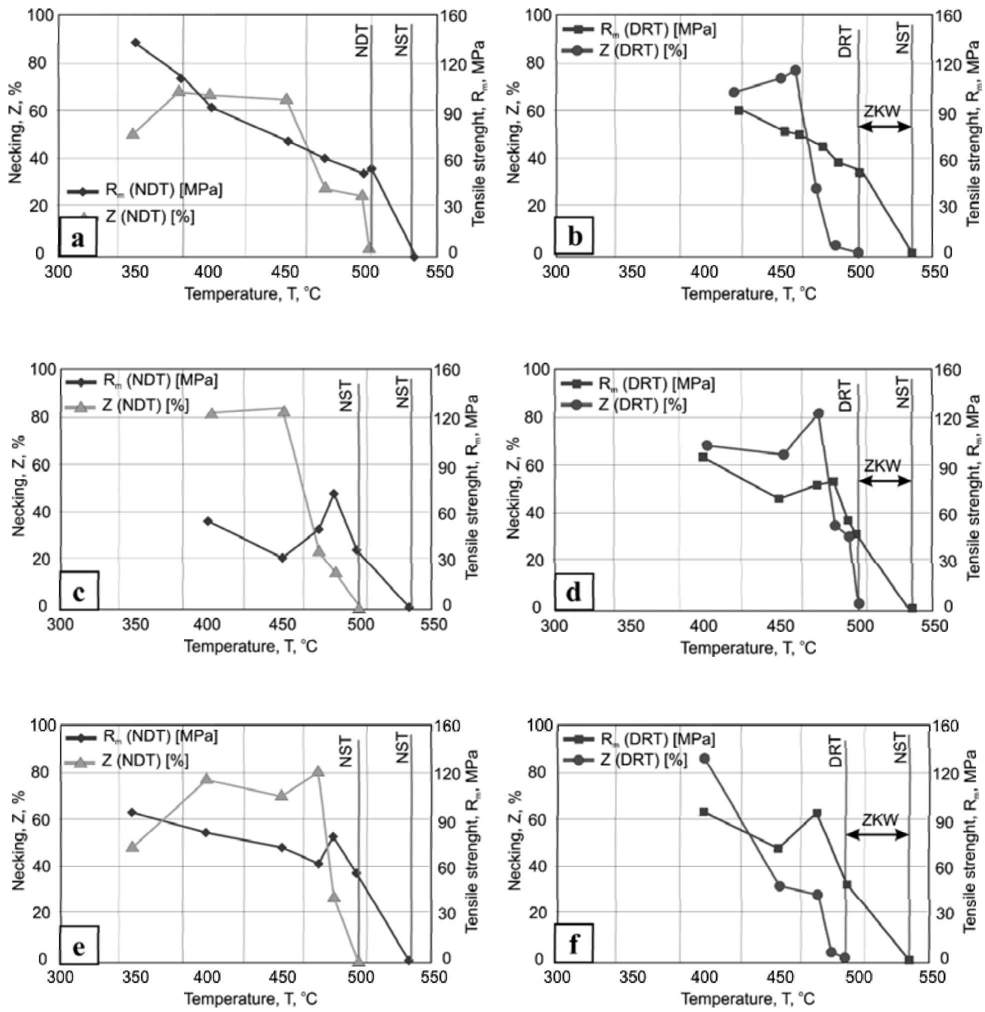

Fig. 7. Contraction and resistance of EN-MCMgRE3Zn2Zr alloy in temperature function: a) material in initial state, heating, NDT test, b) material in initial state, cooling, DRT test, c) material after stress relief annealing (OC I), heating, NDT test, d) material after stress relief annealing (OC I), cooling, DRT test, e) material after stress relief annealing (OC II), heating, NDT test, f) material after stress relief annealing (OC II), cooling, DRT test

9. Analysis of results

Conducted tests of overlay welding and welding enabled the determination of welding parameters of magnesium alloy with addition of zinc and rare earth elements EN-MCMgRE3Zn2Zr. It was stated that it can be welded with TIG method, but a wire with similar chemical composition to the base material should be used as filler metal. Welding or padding should be conducted with alternating current with intensity of 130A with voltage of about 14V, in shroud of pure argon with flow of 14 l/min (table 2).

Conducted tests of padded welds and butt joints microstructures have shown the characteristic structures for welded joints. Parent material was characterised with dendrites of solid solution Mg (α) with inter-metallic phase division $(Mg,Zn)_{12}RE$ [5] (Fig.4). On fusion line, a significant disintegration of structure was found, the crystals of weld were growing on the partially melted basic material grains (Ffig. 4b). In weld area a huge disintegration of grains was found (Fig. 4a).

No influence of material heat treatment before welding on the hardness of the joint was found (fig. 5). The hardness of the welded joint area in all cases did not exceed 65 HV. The crack, found in the area of padded weld in delivery state shows, that it is justified to conduct repairs of casts made of EN-MCMgRE3Zn2Zr alloy after stress relief annealing.

Research on the melting process of EN-MCMgRE3Zn2Zr alloy has shown that in temperature range from 570°C to 616°C, a fusion of inter-metallic phase $(Mg, Zn)_{12}RE$ and the beginning of the melting of crystals of solid solution ( ) occur, and as the liquidus temperature should be assumed with value of 697°C. The heat flow analysis during crystallisation shows, that the crystals of solid solution crystallise in temperature range from 644°C to 598°C, and phase (Mg, Zn)$_{12}$RE begins to crystallise in temperature of 572°C. Solidus temperature equals 552°C.

Temperature NST for alloy EN-MCMgRE3Zn2Zr is 535°, both for material in delivery state and after heat treatment. Hot crack develops in melted inter-metallic phase (Mg, Zn)$_{12}$RE in form of a network along crystals boundaries (Fig. 6a, c, e). The presence of this phase was observed on fracture surface in inter-dendrite areas (Fig. 6b,d,f).

Analysis of NDT test results for EN-MCMgRE3Zn2Zr alloy shows, that ductility loss occurs in 500°C during deformation at a speed of 20 mm/s, independently from heat treatment (table 4). In material in NDT temperature a fusion of phase (Mg, Zn)12RE occurs along crystals boundaries of solid solution and, as a result of disruption of liquid film, a crack appears (fig. 6a,c,e). Melted phase (Mg, Zn)$_{12}$RE crystallises next on the surface of crystals ( ) (Fig.6b,d,f).

The values of resistance to cracking coefficient (from 0.35 to 0,37 for all states of material) also show the lack of influence of heat treatment on NDT temperature (table 4).

Inclination of resistance and plasticity curve in temperature function has a big gradient in dependence to temperature, which proves big resistance to hot cracking (Fig. 7a,c,e). Range between temperature NST and NDT in all cases does not exceed 15°C (Fig. 7a,c,e). Therefore, no influence of heat treatment and deformation speed was proved on the properties of EN-MCMgRE3Zn2Zr alloy in range of crystallisation temperatures.

Tests of the mechanism of alloy EN-MCMgRE3Zn2Zr cracking in HTBR during cooling in DRT tests prove the similar material damage types as in case of heating. Hot cracks formulate as a result of disruption of liquid film on crystals boundaries of solid solution Mg(α). It can be assumed then, that HTBR for this alloy is on constant level between 25°C and 45°C (table 4). No influence of heat treatment on susceptibility to hot cracking was confirmed. The resistance to hot cracking of EN-MCMgRE3Zn2Zr alloy is also confirmed by the big resistance and plasticity gradient in temperature(Fig. 7b,d,f).

10. Conclusions:

On the basis of the conducted tests and results analyses, the following conclusions were drawn:

- EN-MCMgRE3Zn2Zr alloy can be treated as well-weldable with the use of TIG method, independently from the heat treatment. Wires with chemical composition similar to base material should be used as filler metal. Welding or overlay welding should be conducted with the use of alternating current, with intensity up to 130A, by voltage of about 14 V, in shroud of pure argon with flow of 14 l/min (table 3).
- Characteristic temperatures in high-temperature brittleness range were determined for EN-MCMgRE3Zn2Zr alloy: nil-strength temperature NST is 535°C, nil-ductility temperature NDT and ductility recovery temperature DRT equals 500°C. High-temperature brittleness range is in the range from 535-500°C.
- No influence of heat treatment on susceptibility to hot cracking was found. The resistance to hot cracking of EN-MCMgRE3Zn2Zr alloy is also confirmed by the big resistance and plasticity gradient in temperature and constant coefficient of resistance to hot cracking which equals 0.37.

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