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Magnesium and Magnesium Alloys

-- Soluble Magnesium Alloy Materials for Downhole Tools

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Foreword

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IMS holds international conferences on magnesium and supports the publication and presentation of scientific results. Journal of Magnesium and Alloys is the official journal for IMS.

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IMS Standards aim to serve the producing, testing, evaluations, and trades of the global magnesium and magnesium alloy industry, offer standard basis for multiple parties in the industrial chain, intensified technical requirements, and simplify negotiation processes. In the preparation of IMS standard, numbers of relevant data are referred, and the essence contents are extracted, then the targeted modifications are carried out according to the actual situation of magnesium industry. This standard is free on trial, and any parties of magnesium chain are sincerely invited to put forward amendments and suggestions for this standard, especially the technical contents. Please provide amendments and reasons, attaching the necessary proof issues, if possible.

Any feedback or questions on this document should be directed to the secretariat of the Information Committee on IMS (Email: international_Mg@163.com)

Introduction

This standard applies to cast or extruded rod materials of soluble magnesium alloys for petroleum and natural gas industry downhole tools (e.g., fracturing balls, bridge plugs, etc.).

Magnesium and Magnesium Alloys

-- Soluble Magnesium Alloy Materials for Downhole Tools

1 Scope

This standard specifies the requirements, test methods, inspection rules, as well as marking, packaging, transportation, storage, quality certification, and order contract (or purchase contract) content for magnesium and magnesium alloys soluble magnesium alloy materials used in downhole tools.

This standard applies to cast or extruded rod materials of soluble magnesium alloys for petroleum and natural gas industry downhole tools (e.g., fracturing balls, bridge plugs, etc.).

2 Normative References

The following documents are essential for the application of this document. For dated references, only the dated versions apply. For undated references, the latest versions (including all amendments) apply.

ISO 6892-1, Metallic materials — Tensile testing — Part 1: Method of test at room temperature

ISO 16220:2017, Magnesium and magnesium alloys-Magnesium alloy ingots and castings

ISO 8287-2021, Magnesium and magnesium alloys — Unalloyed magnesium — Chemical composition

GB/T 20967, Non-destructive testing - Visual testing - General principles

GB/T 32792, Packing, marking, transporting and storing of magnesium alloy wrought products

GB/T6519-2013, Ultrasonic inspection of wrought aluminium and magnesium alloy products

GB/T 4297, Inspection method for macrostructure of wrought magnesium alloy products

GB/T37596-2019, Magnesium alloy forgings for aerospace

3 Terms and definitions

Soluble magnesium alloy materials for downhole tools refer to cast or extruded rod materials used in the fabrication of petroleum and natural gas industry downhole tools, which exhibit a tensile strength of no less than 150 MPa and a dissolution rate of at least 5 $mg \cdot cm^{-2} \cdot h^{-1}$ in a 93°C 3% KCl solution.

4 Requirements

4.1 Designation

4.1.1 Alloy Grades and Conditions

The grade and conditions of soluble magnesium alloy materials shall conform to the requirements specified in Table 1. When the purchaser requires other grades or conditions, such specifications shall be negotiated and agreed upon between the supplier and purchaser, and explicitly stated in the order contract (or procurement contract).

Table 1 Alloy grades and conditions

Alloy Grade	***Condition***
IMS-WN54M	*H112*
IMS-VW84N	*H112*
IMS-VN21 M	*Cast*
IMS-WC54M	*H112*
IMS-WN95M	*H112*
IMS-ZN42	*H112*
IMS-AZ911M	*T6*
IMS-CN41M	*Cast*

4.1.2 Product Marking

The designation of soluble magnesium alloy materials shall be expressed in the sequence of product name, standard number, grade, conditions, and specifications. Example designations are as follows:

Example 1:

Extruded rod material with grade IMS-WN54M, H112 conditions, diameter 40 mm, and length 3500 mm shall be designated as:

Extruded Rod IMSxxxx-IMS-WN54M H112-Φ40×3500

Example 2:

Cast rod material with grade VN21M, as-cast conditions, diameter 300 mm, and length 1000 mm shall be designated as:

Cast Rod IMSxxxx-IMS-VN21M-Φ300×1000

4.2 Quality Control

Ingots used for the production of soluble magnesium alloy materials shall meet the chemical composition and macrostructure requirements specified in this standard. Other quality requirements for the ingots shall comply with the provisions of ISO 16220:2017.

4.3 Chemical Composition

The chemical composition of the grades of soluble magnesium alloy materials shall conform to the requirements specified in Table 2.

Table 2 Chemical Composition

Element	Grade	Chemical Composition[a] (wt.%)														
		Mg	Al	Zn	Mn	RE	Zr	Y	Gd	Ca	Si	Fe	Cu	Ni	Other elements[b]	
															Individual	Total
MgYNi	*IMS-WN54M*	Rem	—	—	—	—	—	4.5~6.0	—	—	0.05	0.01	0.02	3.5~5.0	0.02	0.2
MgGdYNiMn	*IMS-VW84N*	Rem	—	—	0.6~1.0	—	—	3.5~5.0	7.9~9.0	—	0.05	0.01	0.02	1.0~3.0	0.02	0.2
MgGdNi	*IMS-VN21M*	Rem	—	—	—	—	—	—	1.2~3.0	—	0.05	0.01	0.02	0.4~1.0	0.02	0.2
MgYCu	*IMS-WC54M*	Rem	—	—	—	—	—	4.5~5.0	—	—	0.02	0.02	3.5~4.8	0.05	0.01	0.1
MgYNiZrCa	*IMS-WN95M*	Rem	0.1~0.5	6.2~7.2	—	0.1~0.5	0.02	0.01	0.02	3.8~4.8	—	0.2	0.1~0.5	6.2~7.2	0.01	0.1~0.5
MgZnNi	*IMS-ZN42*	Rem	—	3.5~4.5	—	—	—	—	—	—	0.02	0.02	0.01	1.5~2.5	0.02	0.3
MgAlZnCuNiMn	*IMS-AZ911M*	Rem	9.0~10.0	1.0~2.0	0.1~1.0	—	—	—	—	—	0.05	0.01	1.0~2.0	0.3~0.5	0.01	0.1

MgCuNi	*IMS-CN41M*	Rem	—	—	—	—	—	—	—	—	0.02	0.02	3.5~4.5	0.5~1.0	0.02	0.2
[a] When a single value is specified for the mass fraction in the table, magnesium represents the minimum limit, while other elements represent the maximum limit. [b] Other elements refer to those whose chemical symbols are listed in the table header but for which no specified limit values are defined in this table.																

4.4 Room-Temperature Tensile Mechanical Properties

The room temperature longitudinal tensile mechanical properties of soluble magnesium alloy materials shall conform to the requirements specified in Table 3. Where the properties exceed those specified in Table 3, they shall be mutually agreed upon by the supplier and purchaser and clearly stated in the purchase order (or contract).

Table 3 Room-Temperature Tensile Mechanical Properties

Alloy Grade	***Condition***	***Tensile Strength Rm (MPa)***	***Proof Strength Rp0.2 (MPa)***	***Elongation after Fracture A (% %***
		No less than		
IMS-WN54M	*H112*	*445*	*215*	*12*
IMS-VW84N	*H112*	*350*	*240*	*6*
IMS-VN21M	*Cast*	*175*	*75*	*3*
IMS-WC54M	*H112*	*225*	*120*	*3*
IMS-WN95M	*H112*	*500*	*460*	*6*
IMS-ZN42	*H112*	*295*	*220*	*8*
IMS-AZ911M	*T6*	*360*	*260*	*4*
IMS-CN41M	*Cast*	*210*	*160*	*12*

4.5 Dissolution Rate

The dissolution rate of soluble magnesium alloy materials shall conform to the requirements specified in Table 4. Where the dissolution rate exceeds those specified in Table 4, it shall be mutually agreed upon by the supplier and purchaser and clearly stated in the purchase order (or contract).

Table 4 Degradation Rate

Alloy Grade	***Condition***	***Test Temperature (℃)***	***Test Medium***	***Dissolution Rate $mg \cdot cm^{-2} \cdot h^{-1}$***
				not less than
IMS-WN54M	*H112*	*93*	*3 wt.% KCl*	*60*
IMS-VW84N	*H112*	*93*	*3 wt.% KCl*	*20*
IMS-VN21M	*Cast*	*93*	*3 wt.% KCl*	*14*
IMS-WC54M	*H112*	*93*	*3 wt.% KCl*	*21*
IMS-	*H112*	*93*	*3 wt.% KCl*	*14*

WN95M				
IMS-ZN42	*H112*	*93*	*3 wt.% KCl*	*36*
IMS-AZ911M	*T6*	*93*	*3 wt.% KCl*	*50*
IMS-CN41M	*Cast*	*93*	*3 wt.% KCl*	*80*

4.6 Macrostructure

4.6.1 Macrostructure specimens of ingots for soluble magnesium alloy production shall not permit defects that disrupt metallic continuity, including cracks, inclusions (containing flux slag), gas pores, primary crystal segregation, pipe shrinkage, or coarse grain rings.

4.6.2 Laps with depth not exceeding half of the diameter negative deviation are permissible in macrostructure specimens of bars. For lap-free bars, requirements shall be mutually agreed upon by the supplier and purchaser and clearly stated in the purchase order (or contract).

4.6.3 If the purchaser requires specifications for macrostructure oxide films, metallic compounds, or manganese compounds, such requirements shall be mutually agreed upon and stated in the purchase order (or contract), and shall conform to Section 3.6 of GB/T 37596-2019.

4.7 Ultrasound Flaw Detection

Ultrasonic testing of bars shall comply with Class A requirements specified in GB/T 6519-2013. If alternative testing classes are required, they shall be mutually agreed upon by the supplier and purchaser and explicitly stated in the purchase order (or contract).

4.8 Surface Quality

The surface of soluble magnesium alloy materials shall be clean and free from defects affecting serviceability, including cracks, corrosion, and embedded foreign matter. Surface defects with depth not exceeding the negative deviation — such as extrusion marks, compression marks, handling marks, blisters, scratches, protrusions, or depressions—are permissible.

5 Test procedure

5.1 Chemical Composition

The chemical composition of soluble magnesium alloy materials shall be conducted in accordance with ISO 8287-2021.

5.2 Room-Temperature Tensile Mechanical Properties

The longitudinal room temperature tensile mechanical properties test method of soluble magnesium alloy material is carried out according to the method specified in ISO 6892-1.

5.3 Dissolution Rate

Dissolution rate tests for soluble magnesium alloy materials are performed in accordance with the methods in the Appendix.

5.4 Macrostructure

Macrostructure testing of soluble magnesium alloy materials shall be performed according to the methods specified in GB/T 4297.

5.5 Ultrasound Flaw Detection

Ultrasonic test methods for rods are in accordance with the methods specified in GB/T 6519-2013.

5.6 Surface Quality

Soluble magnesium alloy material surface quality test according to the method specified in GB/T 20967.

6 Result determination

6.1 Inspection and Acceptance

6.1.1 Soluble magnesium alloy materials shall be inspected by the supplier to ensure that the quality of the materials conforms to the provisions of this standard and the order form (or contract), and fill in the quality certificate.

6.1.2 The purchaser shall inspect the received products in accordance with this standard. If the inspection results do not comply with this standard and the order (or contract), the purchaser shall notify the supplier in writing, and the matter shall be resolved through consultation between the supplier and the purchaser. Objections regarding surface quality and dimensional tolerances shall be raised within one month of receipt of the product, while objections of other nature shall be raised within three months of receipt of the product. In case of arbitration, it shall be determined through consultation between the supplier and the purchaser.

6.2 Batch Formation

Soluble magnesium alloys shall be submitted for acceptance in batches. Each batch shall consist of products of the same grade, condition, specification, melt number, and heat treatment furnace, with no limit on batch weight.

6.3 Inspection Items

Before dispatch, each batch of soluble magnesium alloys shall be inspected for chemical composition, room-temperature tensile mechanical properties, degradation rate, macrostructure, ultrasound flaw detection surface quality.

6.4 Sampling

Sampling of soluble magnesium alloys shall comply with the provisions in Table 5.

Table 5 Sampling Provisions

Inspection Item	***Sampling Provisions***	***Clause Number for Requirements***	***Clause Number for Test Methods***
Chemical Composition	*Sampling shall be conducted in accordance with ISO 8287-2021, with one sample taken per batch*	*4.3*	*5.1*
Room-Temperature Tensile Mechanical Properties	*Sampling shall be conducted at 10% of the batch quantity, but not less than 2 pieces. One specimen shall be cut from the front end of each selected soluble magnesium alloy material.*	*4.4*	*5.2*
Dissolution rate	*Sampling shall be conducted at 10% of the batch quantity, but not less than 2 pieces. One specimen shall be cut from the front end of each selected soluble magnesium alloy material.*	*4.5*	*5.3*
Macrostructure	*In the ingots used for the production of soluble magnesium alloy materials, 10% of the quantity of each batch, not less than 2 pieces of sampling, in the extraction of each piece of the tail end of the cut 1 specimen*	*4.6*	*5.4*
Ultrasound Flaw Detection	*Each piece of soluble magnesium alloy material shall undergo 100% inspection prior to delivery.*	*4.7*	*5.5*
Surface Quality	*Each piece of soluble magnesium alloy material shall undergo 100% inspection prior to delivery.*	*4.8*	*5.6*

6.5 Judgment of Inspection Results

6.5.1 If the chemical composition of any specimen fails to meet the requirements, the entire batch shall be rejected.

6.5.2 When the mechanical properties of any specimen are unsatisfactory, double the number of specimens shall be retested from the same batch. The batch shall be accepted only if all retested specimens meet the requirements. If any specimen fails during retesting, the batch shall be rejected.

6.5.3 If the dissolution rate of any specimen is unsatisfactory, double the number of specimens shall be retested. The batch shall be accepted if all retested specimens comply; otherwise, the batch shall be rejected.

6.5.4 When any specimen fails in macrostructure, it is determined as follows:

a) When failing due to cracks, oxide film, metallic compounds and manganese compound segregation, the lot shall be rejected.

b) Failure due to shrinkage tail, coarse crystal ring, bright ring, into the layer, allowed to unqualified bar cut off a certain length and repeat the test, until qualified.

c) The rest of the bar or piece-by-piece inspection, qualified delivery; or according to the maximum cut tail length of the bar retested to qualified cut tail delivery.

d) When there are other defects, the batch of bars shall be dealt with by negotiation between the supply and demand sides.

6.5.5 If the surface quality is non-compliant, the piece is shall be rejected.

6.5.6 If the ultrasonic inspection fails, the piece is shall be rejected.

7 Marking，Packing，Transportation and storage

7.1 Marking

7.1.1 Marking

The following marks shall be applied to the front end of qualified soluble magnesium alloy products:

a) Grade;

b) Conditions;

c) Specification;

d) Batch number.

7.1.2 Packaging markings

Soluble magnesium alloy material box marking should be consistent with the provisions of GB/T 32792.

7.2 Packaging, Transportation, and Storage

7.2.1 Soluble magnesium alloys shall be packaged with oil coating. Special requirements, if any, shall be agreed upon by the supplier and the purchaser and noted in the order (or contract).

7.2.2 Soluble magnesium alloy material packaging, transportation, storage of other requirements should be consistent with the provisions of GB/T 32792.

7.3 Quality Certificate

Each batch of soluble magnesium alloy material shall be accompanied by a product quality certificate stating:

a) Name, address, telephone number and fax number of the supplier;

b) Product name;

c) Grade number;

d) Conditions;

e) Specification and accuracy level;

f) Lot number;

g) Net weight and number of pieces;

h) the test results of the various analytical items;

i) This standard number;

(j) Factory date (or packaging date).

8 Orders or tenders

Orders (or contracts) for products listed in this standard shall include the following information:

a) Product name;

b) Grade number;

c) Conditions;

d) Dimensions and tolerances;

e) Net weight or quantity;

f) Special requirements of the purchaser:

— Special dimensional tolerance requirements;

— Special tensile mechanical property requirements;

— Special packaging requirements;

— Other special requirements;

g) The number of this standard;

h) Others.

Appendix

(Normative Appendix)

Determination of Dissolution Rate by Weight Loss Method

A.1 Scope: This appendix provides a method for the determination of the dissolution rate of soluble magnesium alloy materials, which is also applicable to the determination of industrial dissolution rates for dissolution purposes.

A.2 Reagents: A KCl solution with a mass fraction of 3%.

A.3 Equipment: Water bath device, temperature control accuracy ≤±1℃.

A.4 Sample Preparation: Each specimen shall be individually placed in a glass container of sufficient size to ensure the reagent fully submerges the specimen. The ratio of reagent volume (cm^3) to the exposed surface area (cm^2) of the specimen shall be no less than 40:1. The specimen shall not contact the glass surfaces, except at line or point contact positions used to secure the specimen.

A.5 Immersion Test Procedure: First, weigh the specimen with an accuracy of 1 mg and measure its exposed surface area. Heat the reagent in the thermostatic bath to 93 ± 1° C, then immerse the specimen in the reagent and seal the test chamber of the bath apparatus to prevent reagent evaporation. Maintain the temperature at 93 ± 1° C throughout the test. At 1-hour intervals, remove the specimen, dry it, re-weigh it, and re-measure its surface area. Repeat this process for 4–10 cycles, calculating the single-cycle dissolution rate (mass loss per unit surface area per hour) after each cycle. The test is terminated when the difference between two consecutive dissolution rates is less than 10% (relative to the prior rate), indicating stabilization. Finally, report the average of the last two dissolution rates as the material's stable dissolution rate.

A.6 Result Calculation: The dissolution rate is obtained using the following formula:

$$W=\triangle m/(S_{avg}\cdot T)$$

W is the dissolution rate, in units of $mg\cdot cm^2\cdot h$;

△m is the mass loss between two consecutive measurements, in units of mg;

S_{avg} is the average surface area between two consecutive measurements, in units of cm^2;

T is the time interval between two consecutive measurements, in units of h; The dissolution rate for each individual cycle is calculated based on a 1h interval.

A.7 Test Report:

The test report shall at least include:

a) Detailed description of the specimen, including chemical composition, alloy grade, etc;

b) Reagent type and concentration, test temperature;

c) Measurement results, including the mass of the sample before and after dissolution, dissolution time, contact area with the test solution, and dissolution rate;

d) Name of the person who made the determination;

e) Date of determination;

f) Signature of the person making the determination.