



Magnesium alloy biomaterials: Innovative engineering solutions for future biodegradable implant materials

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ABSTRACT

Magnesium (Mg) alloys are promising candidates for temporary orthopaedic and cardiovascular implants due to their biocompatibility, mechanical compatibility with bone, and complete biodegradability. Although extensive research has focused on alloying and conventional heat treatments to tailor their performance, the application of deep cryogenic treatment (DCT) remains largely unexplored. In this study, the influence of DCT on the microstructure, mechanical properties, and corrosion behaviour of Mg–Gd alloys with different Gd contents was systematically investigated. Three compositions Mg–2Gd, Mg–5Gd, and Mg–10Gd were examined under conventionally extruded (CHT) and DCT (–196 °C) for 8 h, 24 h and 48 h states. Microstructural and phase characterization was performed using scanning electron microscopy (SEM), X-ray diffraction (XRD) and atom probe tomography (APT). Additionally, XRD-based surface micro-residual stresses were analysed to observe changes in the stress state of the material. Next, corrosion behaviour was evaluated by potentiodynamic polarisation, while Raman shift spectroscopy was used to analyse surface oxides and passivation layer formation. Mechanical response was assessed via Vickers microhardness testing. The results demonstrate a composition-dependent response to DCT. An increase in hardness was observed for Mg–5Gd, whereas Mg–2Gd and Mg–10Gd exhibited reduced hardness following treatment. Changes in corrosion behaviour were also observed for all three alloys after DCT, suggesting that cryogenic processing alters the electrochemical response of the materials, likely through modifications of the microstructure. These findings highlight the potential of DCT as a supplementary processing route for tailoring the performance of biodegradable Mg–Gd alloys for biomedical applications.

1. Introduction

From a materials perspective, magnesium (Mg) alloys possess several intrinsic properties that make it attractive as biomaterials owing to their unique combination of biodegradability, biocompatibility and mechanical properties [1,2]. In particular, the relatively low density (1.738 g cm^{–3}) and elastic modulus of Mg (40–50 GPa), help to reduce stress shielding and promote more physiological load transfer between the implant and surrounding tissue [3]. Conventional implant materials such as stainless steel, cobalt–chromium alloys, and titanium alloys exhibit excellent mechanical strength and corrosion resistance.

However, their permanent nature often leads to long-term complications, including stress shielding, chronic inflammation, and the need for secondary surgical procedures [3]. As a result, research has increasingly focused on biodegradable materials especially for cardiovascular stents, among which Mg alloys have emerged as particularly promising candidates. Despite these advantages, the biomedical applications of Mg alloys remain limited by several critical challenges. Pure magnesium exhibits insufficient mechanical strength and exhibits excessively high corrosion rate under physiological conditions, leading to rapid loss of mechanical integrity, localized alkalization and hydrogen gas evolution [4–6]. In cardiovascular stent applications, uncontrolled degradation

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can result in premature collapse of the scaffold, compromising vessel support before complete healing and remodelling occur [7,8]. Therefore, improving both the mechanical performance and corrosion resistance of Mg alloys is essential for their successful use as biodegradable implants.

Alloying has proven to be one of the most effective strategies for enhancing the properties of Mg. Elements such as Zn, Ca and rare-earth elements (REEs) have been extensively investigated due to their ability to improve strength, ductility, and corrosion behaviour through solid solution strengthening and precipitation hardening mechanisms [9]. Among REEs, Gd [10,11] has attracted increasing interest because of its high solubility in Mg, strong precipitation-strengthening effect, and relatively low cytotoxicity at controlled concentrations [12,13]. Mg-Gd-based alloys have demonstrated superior mechanical properties compared to many conventional Mg alloys, including improved tensile strength and thermal stability, while also offering potential for controlled degradation behaviour [14–18]. However, achieving an optimal balance between mechanical integrity and degradation rate remains a key challenge for Mg-Gd alloys intended for such applications. To further optimize the performance of Mg alloys, advanced processing techniques beyond conventional alloying and heat treatment are being explored. Deep cryogenic treatment (DCT) is one of these advanced processing techniques that involves exposing materials to extremely low temperatures, typically using liquid nitrogen as a cooling media at approximately -196°C , for extended periods. The underlying mechanism of DCT is associated with microstructural refinement, increased dislocation density, redistribution or refinement of phases, and stabilization of precipitates, which can significantly influence material properties [19–22]. To date, research on the influence of DCT on Mg-Gd alloys has not been well understood. Nevertheless, existing investigations on Mg alloy systems have demonstrated that DCT can lead to increased hardness and strength, primarily through α -Mg grain refinement and changes in β -phase precipitate morphology and distribution [23,24]. Similar improvements have been reported for REE-containing magnesium alloys, such as WE43, for which DCT combined with aging treatment resulted in increased hardness, improved wear resistance, and enhanced micromechanical properties [25]. These findings suggest that DCT can work synergistically with alloy composition and heat treatment to further improve Mg alloys. In addition to mechanical enhancement, DCT has been reported to influence the corrosion behaviour of Mg alloys, which is of particular importance for biomedical applications [26]. DCT can alter the morphology, size, and distribution of second-phase particles, thereby reducing micro-galvanic coupling between the matrix and secondary phases [21,24,27]. As a result, several studies have observed improved corrosion resistance and more uniform degradation behaviour in cryogenically treated magnesium alloys [19,26,28,29]. For example, DCT of Mg-Y-Nd alloys was shown to reduce corrosion rates and improve electrochemical stability compared to conventionally treated counterparts, with microstructural refinement identified as the primary contributing factor [21]. Similar trends have been observed in Mg-Zn-Ca and Mg-Y-Nd systems, indicating that DCT has broad potential for tailoring corrosion performance in variety of biodegradable Mg alloys [21,27,30].

Given the stringent performance requirements of biodegradable implants—particularly cardiovascular stents, where both mechanical reliability and controlled degradation are essential—novel processing routes are needed to further enhance the performance of Mg alloys. In this work, we investigate the effect of deep cryogenic treatment (DCT) on three Mg-Gd alloys with varying Gd contents (Mg-2Gd, Mg-5Gd, and Mg-10Gd), aiming to improve their mechanical and corrosion properties for biomedical applications by tailoring the microstructure and surface micro-residual stresses. By systematically analysing the influence of DCT on microstructure, microhardness, micro-residual stresses and corrosion resistance, this study seeks to provide deeper insight into the potential of DCT as an effective tool for optimizing Mg-Gd alloys for biodegradable implant applications.

2. Material and methods

2.1. Material, processing, microstructure and phase analysis

In this study, as extruded Mg-Gd alloys with Gd contents of 2 wt%, 5 wt% and 10 wt% were investigated. The material was produced at the Institute of Metallic Biomaterials (Hereon), Geesthacht, Germany, as described by Harmuth et al. [17] with an extrusion temperature of 400°C and a ram speed of 2.2 mm/s (profile exit speed 3.0 m/min). The chemical composition measured by X-ray fluorescence is given in Table 1.

DCT was performed using liquid nitrogen (-196°C). The specimens were cooled to the cryogenic temperature and subsequently reheated at an approximate rate of $10^{\circ}\text{C min}^{-1}$, with soaking times of 8 h, 24 h and 48 h.

A Zeiss Crossbeam Merlin I scanning electron microscope (SEM) with an integrated EDAX Velocity EBSD camera were used to perform a microstructural analysis of the Mg-Gd alloys at MPI SusMat. Quantitative phase analysis was performed using a Rigaku SmartLab diffractometer with a Co K α X-ray source. This diffractometer is equipped with micro-area optics ($500\text{ }\mu\text{m} \times 500\text{ }\mu\text{m}$ beam size), a 5-circle goniometer, and a HyPix 3000 area detector. Measurements were performed using a continuous, symmetric overview scan with a step size of $\Delta 2\theta = 0.01^{\circ}$, a count rate of $4^{\circ}/\text{min}$, and a power setup of 45 kV/200 mA. Rietveld simulation was then employed to quantify each phase from the measured data.

2.2. Atom probe tomography (APT)

Atom probe tomography (APT) specimens were prepared using a Ga-Focused Ion Beam (FIB) (Helios 5CX, Thermo Fisher Scientific) following a standard sample preparation protocol from Thompson et al. [31]. APT experiments were performed using a Cameca LEAP 5000 XR (reflectron) atom probe. APT measurement was performed in voltage pulsing mode with a pulse fraction of 15%, a repetition rate of 125 kHz at a base temperature of 40K with a target detection rate of 0.5 %. A total of three samples were measured for DCT and CHT Mg5Gd. Crystallographic poles on the detector were used to determine the field and image compression factors and to spatially-calibrate the reconstruction. Data reconstruction was performed using Cameca's Integrated Visualisation and Analysis Software (IVAS) in APT Suite 6.3.

2.3. Thermodynamic modelling

Thermodynamic modelling was conducted in order to understand ratio and formation of phases during exposure to [cryogenic temperatures](#) versus no DCT. It was performed with Thermo-Calc Software using TC2025b version and the TCMG8 database.

2.4. Surface micro-residual stress measurements

Surface micro-residual stress measurements were conducted on specimens with the same dimensions as those prepared for compressive strength testing. X-ray diffraction (XRD; Rigaku SmartLab 6 kW) with Co K α X-ray source was carried out at an operating voltage of 45 kV and a current of 200 mA. The scans were collected over a $0-45-90^{\circ}$ range with a specimen-to-detector distance of 149.36 mm. The surface residual stresses were measured in all three directions, as it is standard practice to provide the minimum data required to calculate the state of stress on the material's surface. The 0 axis measures the strain along the primary reference axis (longitudinal direction), the 45 axis measures the diagonal (shear) deformation between 0 and 90° , and the 90 axis measures the strain perpendicular to the primary axis (transverse direction). Correlating all the measurements helps to understand changes in stress (tensile vs compressive in a 2D surface manner) correlated to changes in the microstructure (i.e. phase changes, recrystallization and precipitation).

Table 1

Chemical composition of three types of Mg–Gd alloys: Mg–2Gd, Mg–5Gd, and Mg–10Gd. The Gd content was measured using X-ray fluorescence, while the remaining elements were analysed by atomic absorption spectroscopy.

Alloy	Gd wt. %	Fe wt. %	Cu wt. %	Ni wt. %	Al wt. %
Mg-2Gd	1.75 ± 0.05	0.0038 ± 0.0006	0.0016 ± 0.0001	0.0004 ± 0.0001	<0.015
Mg-5Gd	4.66 ± 0.04	0.0054 ± 0.0003	0.0045 ± 0.0002	0.0006 ± 0.0001	<0.015
Mg-10Gd	9.48 ± 0.47	0.0129 ± 0.0013	0.0030 ± 0.0001	0.0017 ± 0.0001	<0.015

Quantitative evaluation of residual stress was performed using Bruker TOPAS V5.0 software. The procedure for surface micro-residual stress determination followed the methodology reported [32].

2.5. Microhardness (Vickers)

The microhardness of the heat-treated samples was determined at room conditions (21 °C and 50% relative humidity) using the Vickers HV0.1 method at MPI SusMat, Germany. For each specimen, at least six indentations were made, and the average value and standard deviation (SD) were calculated. Statistical analysis of the collected data was carried out using SPSS software (PASW Statistics 18, SPSS Inc., USA), while post-processing and data visualisation were performed with Origin 2025 (OriginLab Corporation, USA).

2.6. Potentiodynamic measurements

Electrochemical characterization was conducted with a Vertex One potentiostat (Ivium Technologies, Netherlands) at MPI SusMat. Metallographically prepared specimens (grinded and polished down with 1 µm) were mounted in a corrosion cell, where the exposed working surface (0.283 cm²) was defined by a sealing O-ring with a 6 mm diameter. Measurements were performed in a 0.9% NaCl solution under laboratory ambient conditions (21 °C, 50 % relative humidity). Before electrochemical testing, the working electrode was allowed to stabilize at its open circuit potential (OCP) for 5 min. Electrochemical impedance spectroscopy (EIS) was performed at OCP in the frequency range of 0.1 Hz–100 kHz using an input voltage of amplitude 10 mV followed by 10 min cathodic cleaning at −1.0 V vs. Ag/AgCl. After a subsequent 1 h OCP period. This was followed by cyclic voltammetry (CV) from −500 mV vs Ag/AgCl until a current density of 3.5 mA/cm² was reached. The testing sequence was based on commonly used ASTM corrosion-testing practices. Corrosion potential and corrosion current density values were obtained from polarisation curves using Tafel analysis.

2.7. Raman shift spectroscopy

Raman analysis was performed at MPI SusMat using a LabRAM confocal Raman microscope (Horiba Jobin Yvon, France) equipped with a CCD detector, a 600 L mm^{−1} diffraction grating, and a 50× objective lens. Excitation was provided by a 532 nm HeNe laser, which was focused onto an area of approximately 10 µm² on the specimen surface. The applied laser power at the sample was maintained between about 3 and 4 mW. Spectral acquisition was carried out using three accumulations of 600 s each, yielding a spectral resolution of approximately 1 cm^{−1}. Oxide phases were identified by comparing the measured spectra with reference data available in the RRUFF Raman spectral database.

3. Results and discussion

3.1. Exploring the effect of DCT on microstructure wrought Mg–Gd alloy with correlation to modelling-microstructure

XRD analysis confirms that, in all samples, the dominant phase is the α-Mg hexagonal close-packed (HCP) matrix with minor intermetallic phases, and that the phase fractions are governed primarily by alloy

composition rather than cryogenic exposure (Fig. 1a–c, Table 2). Mg-2Gd alloy contains approximately 0.9 vol% Mg₅Gd in the CHT condition. After DCT, only minor variations are observed. Mg₅Gd measures about 1.4 vol% after DCT-8 h, decreases to approximately 0.5 vol% after DCT-24 h, and increases again to around 0.9 vol% after DCT-48 h. For Mg-5Gd, the intermetallic fraction remains essentially unchanged across all conditions. Mg₅Gd varies only slightly between about 1.2 vol% and 1.6 vol% following DCT-8 h, DCT-24 h, and DCT-48 h treatments. In Mg-10Gd, the Mg₅Gd intermetallic phase shows a gradual increase with extended cryogenic duration. It rises from about 2.1 vol% in the CHT condition to approximately 2.2 vol% after DCT-8 h, 2.3% after DCT-24 h, and 3.0% after DCT-48 h.

No additional diffraction peaks emerge following DCT, indicating that cryogenic treatment does not induce new crystalline phase formation detectable by XRD. The phase stability observed after DCT is consistent with the diffusion-controlled precipitation behaviour of Mg–Gd alloys [33]. Intermetallic phase evolution in these systems occurs predominantly during elevated-temperature processing, where sufficient atomic mobility enables solute redistribution and precipitate growth. At cryogenic temperature, substitutional diffusion of Gd in the Mg matrix is severely restricted. Therefore, in our case, DCT does not alter the equilibrium phase constitution, but instead it is expected that DCT affects the defect structure and lattice strain state established during prior thermal history [12,34]. In this context, DCT should be interpreted as a strain-mediated microstructural tuning process occurring under diffusion-limited conditions, rather than a precipitation-driven transformation.

XRD also reveals a change in the crystallographic features that are markedly displayed by the relative intensity change of the main diffraction peaks related to {10 $\bar{1}$ 0}, {0002} and {10 $\bar{1}$ 1} as presented by the enlargements in Fig. 1a–c. The insets highlight the 37–43° 2θ region, where peak intensity rises from Mg-2Gd to Mg-5Gd, while Mg-10Gd exhibits slightly higher intensity than Mg-2Gd, although not in a strictly linear fashion. These changes take place within the existing α-Mg matrix and show a dependency also based on the Gd content. Such texture modifications are previously observed in REE-containing Mg alloys [35,36]. However, the variation in the intensity of the aforementioned peaks with DCT is nonlinear across the compositions and becomes more pronounced in Mg-5Gd and Mg-10Gd. This suggests a potential lattice-scale strain redistribution rather than a structural transformation that is influenced by the higher presence of Gd solutes. These observations also indicate a potential influence of DCT on the crystallography and local lattice strains within the existing α-Mg matrix, which will be further explored with electron backscatter diffraction (EBSD).

Thermodynamic modelling (Fig. 1d–f) supports the experimental observations and clarify the compositional dependence of intermetallic stability. In Mg-2Gd, Mg₅Gd appears with a small fraction (~0.02) at low temperature and dissolves near ~60 °C, while the HCP remains dominant until melting (~630 °C). In Mg-5Gd, Mg₅Gd persists to ~170 °C with a slightly larger fraction (~0.025), whereas in Mg-10Gd the intermetallic fraction increases further (~0.1 at cryogenic temperature) and remains stable up to ~300 °C. These results demonstrate the increasing thermodynamic stability of Mg₅Gd with rising Gd concentration and explain the composition-dependent intermetallic fractions measured by XRD. The agreement between thermodynamic modelling

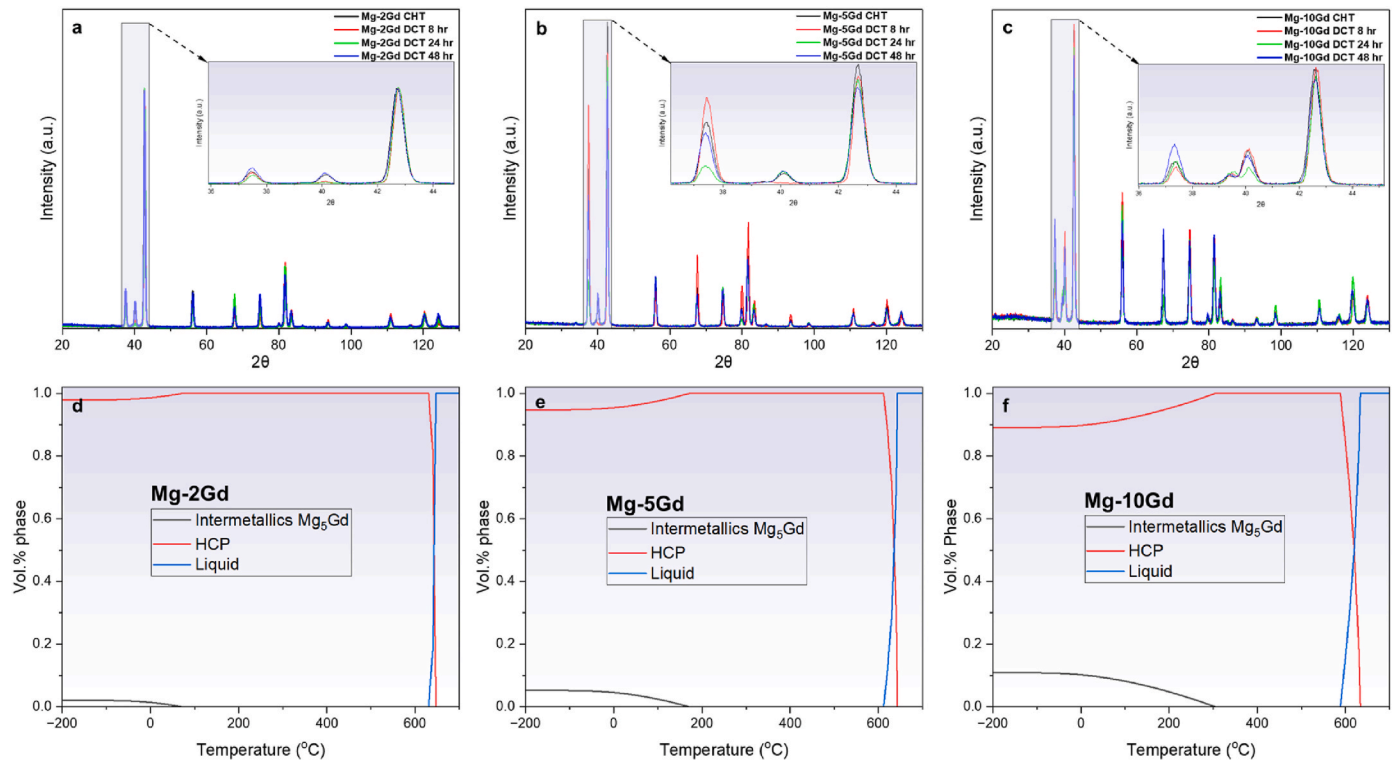


Fig. 1. (a–c) Stacked XRD patterns of Mg-2Gd, Mg-5Gd, and Mg-10Gd alloys in CHT, DCT-8 h, 24 h, and 48 h conditions. (d–f) Thermo-Calc phase diagrams of Mg-2Gd, Mg-5Gd, and Mg-10Gd alloys, respectively.

Table 2

XRD-identified phases in Mg-2Gd, Mg-5Gd, and Mg-10Gd alloys following CHT (extruded), DCT 8 h, 24 h and 48 h.

Alloy	Condition	vol. % α -Mg (HCP)	vol% $Mg_{5}Gd$ (FCC)
Mg-2Gd	CHT	99.1	0.9
	DCT 8 h	98.6	1.4
	DCT 24 h	99.5	0.5
	DCT 48 h	99.1	0.9
Mg-5Gd	CHT	98.4	1.6
	DCT 8 h	98.8	1.2
	DCT 24 h	98.4	1.6
	DCT 48 h	98.5	1.5
Mg-10Gd	CHT	97.9	2.1
	DCT 8 h	97.8	2.2
	DCT 24 h	97.7	2.3
	DCT 48 h	97.0	3.0

and experimental diffraction data indicates that the intermetallic phases are stable well below room temperature. Thus, the data suggests that DCT occurs within an established phase field, and the observed cryogenic response is governed by defect evolution and lattice strain redistribution rather than by phase transformation [13,37].

EBSD revealed pronounced, composition-dependent microstructural responses to DCT (Figs. 2, 6 and 7). In Mg-2Gd, inverse pole figure (IPF) maps showed relatively coarse grains and limited twinning in the CHT state, while DCT-24 h induced noticeable grain refinement and enhanced twinning, suggesting that cryogenic treatment promotes twinning through lattice contraction. Phase mapping combined with energy-dispersive X-ray spectroscopy (EDS) maps confirmed HCP matrix dominance with minor body-centred cubic (BCC) intermetallics, corresponding to $Mg_{5}Gd$ inferred from XRD. Kernel average misorientation (KAM) maps indicated an increase in local misorientation, reflecting elevated internal strain likely arising from lattice contraction and defect redistribution. Grain boundary analysis further highlighted increased fractions of low and medium angle boundaries, indicative of twinning-

assisted grain subdivision and microstructural homogenization [26, 38]. However, the samples display also a considerable amount of retained deformation on the surface as a preparation artefact, which was identified through KAM and high grain fragmentation as presented in Fig. 2f–h. Consequentially, a routine with light etching with picric acid was performed to remove the surface preparation artefacts and to elevate the microstructural features related to crystallography and twinning. Example of the effect of etching and twinning identification for CHT and DCT samples is presented in Fig. S1.

As seen from Fig. 3, the twins are present in both CHT and DCT samples, but respectively show a large difference in the morphology and nucleation patterns. While DCT shows longer and more pronounced twins, the CHT sample displays more fractionated twins. This can be potentially related to the less homogenized stress state of the CHT material in comparison to the DCT one. However, the presence of twins in the microstructure is highly sporadic and site-dependent, but overall appears to increase with the application of DCT and with longer cryogenic exposure times. To confirm this in a more statistically-relevant manner, low-magnification EBSD was performed at which the main emphasis was given on understanding the correlation of DCT time on the twin formation. For this reason, these measurements were performed on DCT samples with 8 h and 48 h exposure [39].

The results as given in Figs. 3–5, indicate a clear difference for the Mg-2Gd case, which displays considerably higher amount of twins development after 48 h of DCT vs the 8 h one (compare Fig. 3a with 3d). This was quantitated using grain boundary analysis, as presented in Fig. 3b and e), which indicates a nearly 3-fold increased twinning formation with the longest DCT exposure compared to the 8 h case. The formation of twins also reduces the average grain size and increases the grain size distribution (by about 10%), which can be seen from comparing Fig. 3c with 3f, due to the segmentation of the overall microstructure. It should be noted that through the grain boundary analysis (example Fig. 3j), the compression twins (with the usual 56° misorientation) are excluded from the twinning analysis due to the overlap with regular grain boundaries and thus we identify the induced

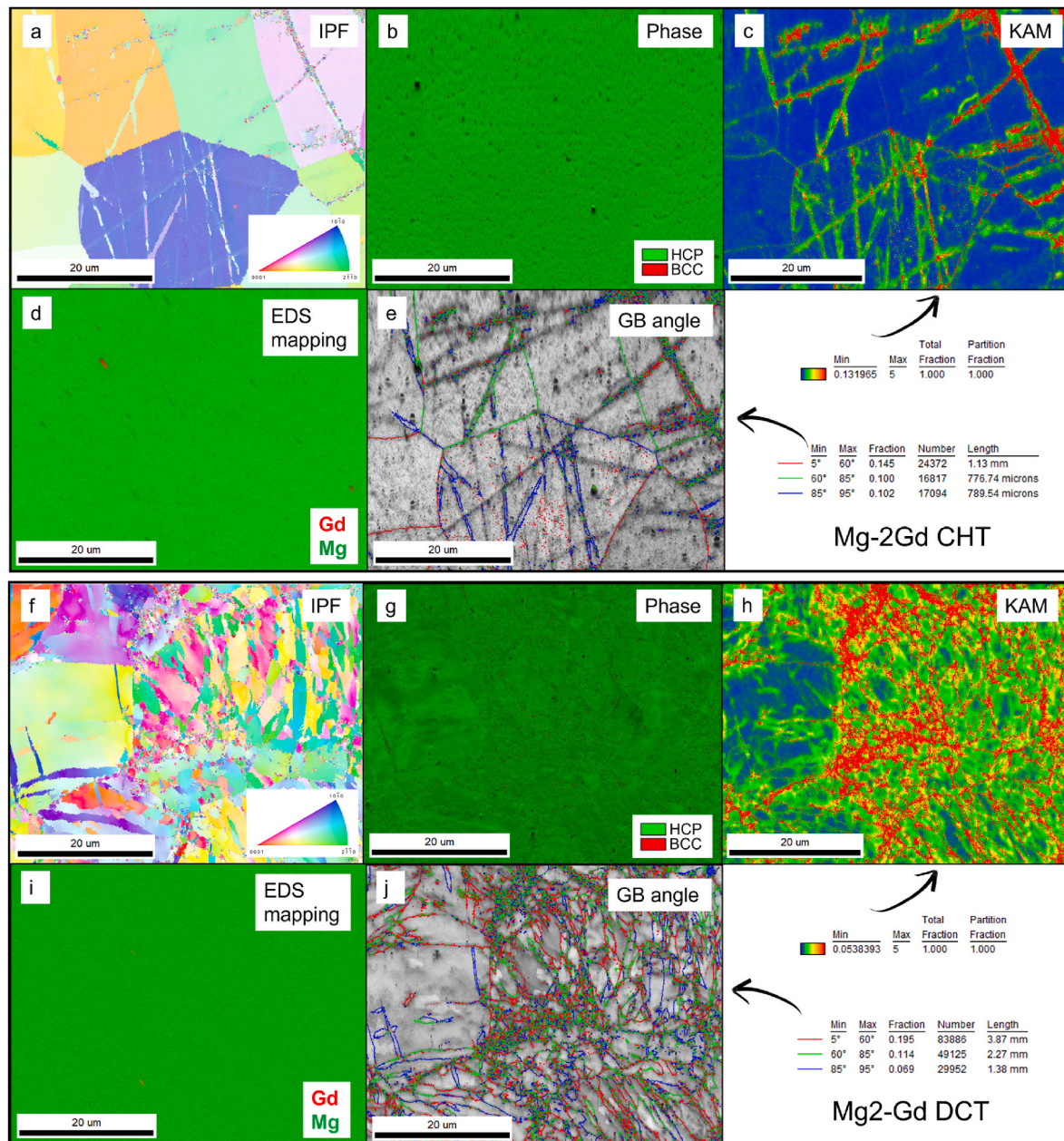


Fig. 2. Microstructural characterization of the Mg-2Gd alloy in the CHT (extruded) and DCT-24 h conditions. (a-e) Characterization of the CHT condition: (a) inverse pole figure (IPF) map showing grain orientation distribution, (b) Phase identification map confirming HCP Mg as the dominant phase with Mg₂Gd as a minor phase, (c) kernel average misorientation (KAM) map indicating local strain distribution, (d) EDS elemental mapping illustrating the spatial distribution of Mg and Gd, and (e) grain boundary misorientation angle distribution. (f-j) Characterization of the DCT-24 h condition in Mg-2Gd: (f) IPF map, (g) phase identification map, (h) KAM map, (i) EDS elemental mapping, and (j) grain boundary misorientation angle distribution, showing the microstructural evolution.

twinning only through the tensile twins (commonly forming with $\{10\bar{1}2\}$ planes) expected to have an average 90° misorientation to the parent grains [39]. The pole figure projections (see Fig. 3g and h), along the selected directions also portray the clear texture of the microstructure at nearly $\{10\bar{1}0\}$, induced by the extrusion process, as well as formation of twins. The pole figure projections also can identify the presence of the compressive twins (when comparing the $\{10\bar{1}1\}$, $\{10\bar{1}2\}$ and $\{10\bar{1}3\}$ projections with the $\{10\bar{1}0\}$ projection) and separate them from the tensile twins. In this regard, Fig. 3g, indicates a simultaneous formation of both compressive and tensile twins in the case of CHT, which follows the texture dependency with the texture. However, when DCT is applied, the twins facilitate a clearer activation that is hallmarked by the pronounced ring-like development in the $\{10\bar{1}2\}$ projection. In

turn, the other twinning projections show no concrete difference to the CHT sample. This stipulates that despite the similar texture, the DCT facilitates higher twinning, which is predominantly related to the tensile twin character as confirmed from the IPF mapping in Fig. 3e.

With higher alloying content, the Mg-5Gd and Mg-10Gd alloys present a progressively less defined texture compared to the Mg-2Gd alloy as well as increasingly larger average grain size with alloying content (approximately $25\ \mu\text{m}$ and $29\ \mu\text{m}$ for Mg-5Gd and Mg-10Gd, respectively), regardless of the [40] CHT or DCT state (compare Figs. 3 and 4 and 5). The loss of texturing is related to the predominant effect of dynamic recrystallization (DRX) during the extrusion process that is assisted by the higher Gd content to facilitate then faster kinetics of the DRX process as observed for lower Gd content [40]. Additionally, higher Gd content facilitates the formation of the so-called RE texture that

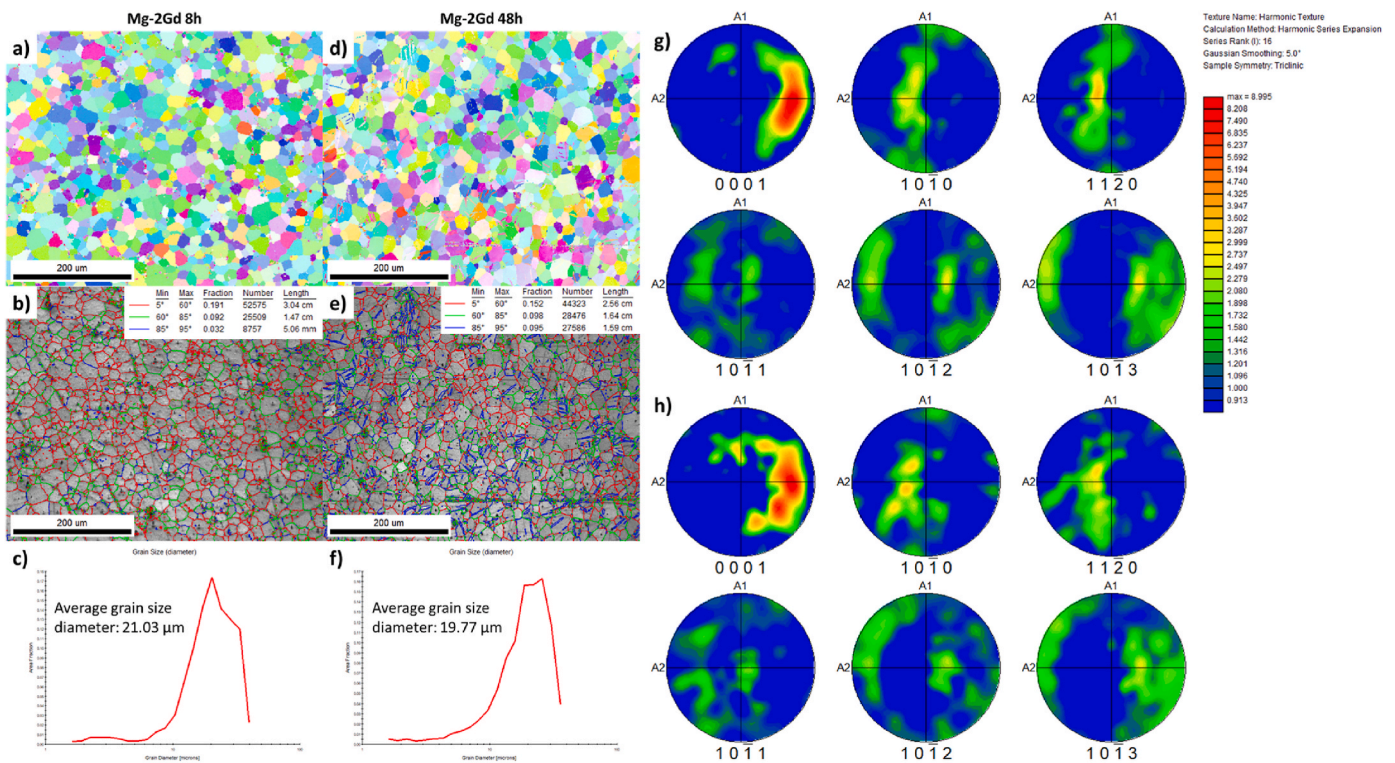


Fig. 3. EBSD-determined information on the crystallographic features of the Mg-2Gd alloy with 8 h (a-c, g) and 48 h (d-f, h) of DCT. a and d present the IPF mapping, b and e the grain boundary analysis, c and f the grain size distribution and g and h present the IPF projection maps. A1 direction coincides approximately with the transversal direction and the A2 with the extrusion direction.

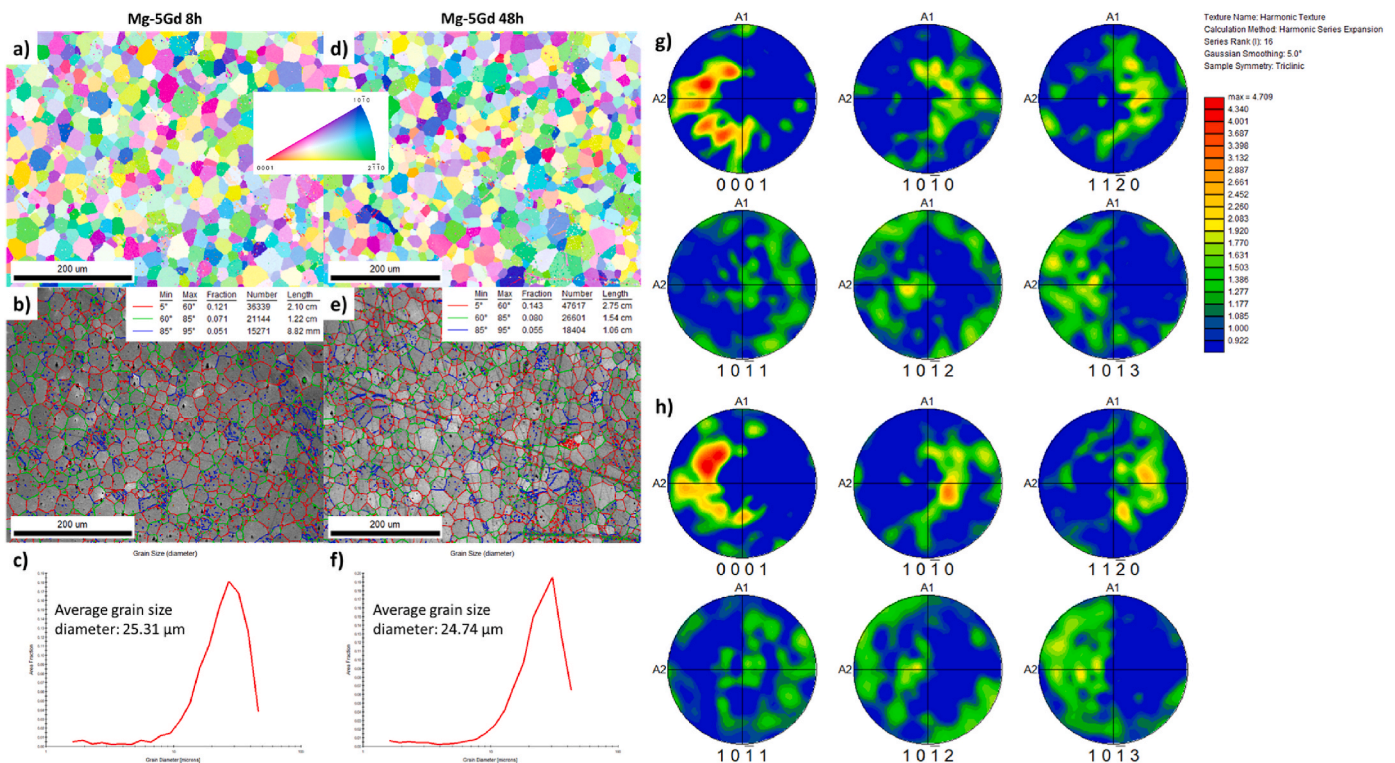


Fig. 4. EBSD-determined information on the crystallographic features of the Mg-5Gd alloy with 8 h (a-c, g) and 48 h (d-f, h) of DCT. a and d present the IPF mapping, b and e the grain boundary analysis, c and f the grain size distribution and g and h present the IPF projection maps. A1 direction coincides approximately with the transversal direction and the A2 with the extrusion direction.

results from the solute dragging effect [41]. Resultingly, the increased grain size is considered to be primarily related to secondary

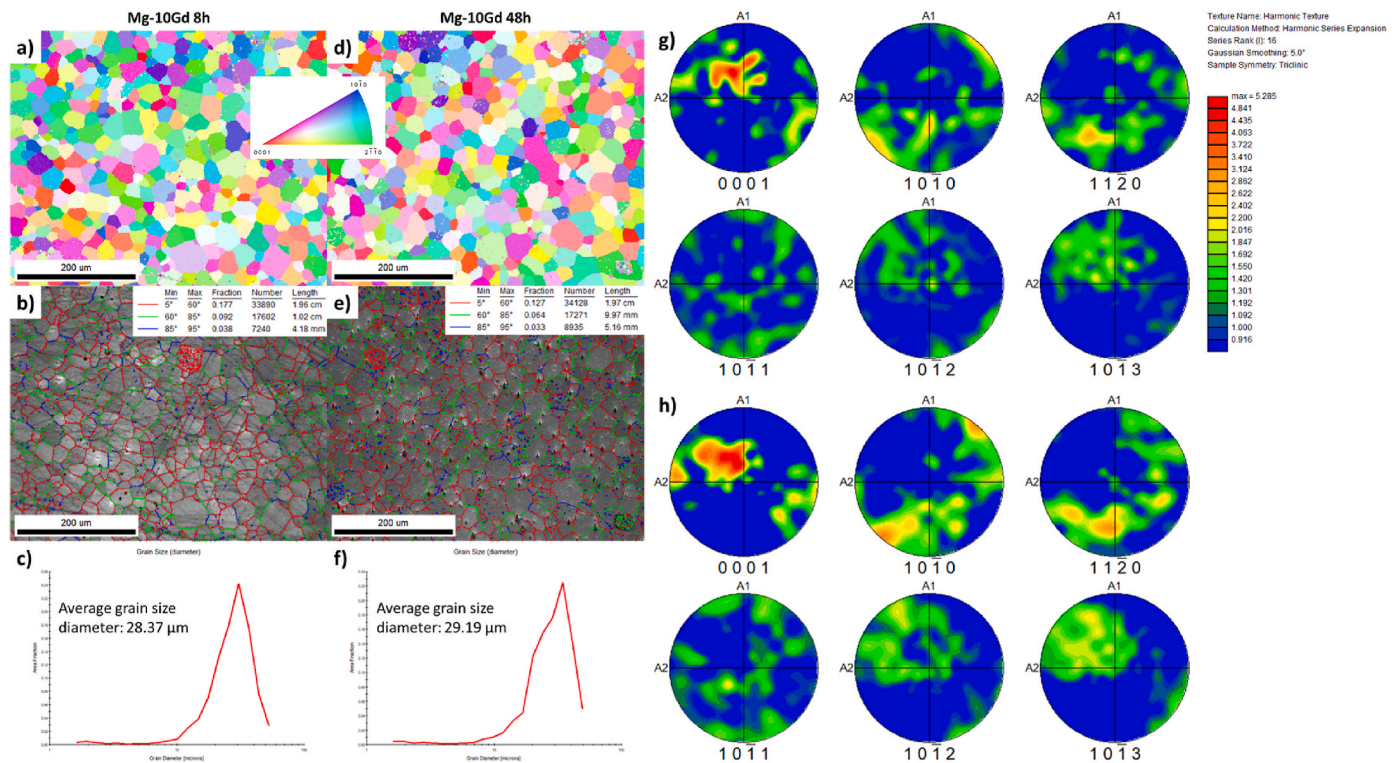


Fig. 5. EBSD-determined information on the crystallographic features of the Mg-10Gd alloy with 8 h (a-c, g) and 48 h (d-f, h) of DCT. a and d present the IPF mapping, b and e the grain boundary analysis, c and f the grain size distribution and g and h present the IPF projection maps. A1 direction coincides approximately with the transversal direction and the A2 with the extrusion direction.

recrystallization, i.e. grain growth, as expected for higher temperature extrusion of Mg alloys, when sufficient thermal energy exists in the final billet that simulate the latter stages of what is observed for static recrystallization [42]. This in turn is also noticed by the reduced effect of DCT on the twinning phenomena. While the twinning enhancement is still pronounced for the case of Mg-5Gd with about 20 % more in the 48 h DCT case against the 8 h one. The overall fraction of the twins is also reduced for both CHT and DCT cases, at which the Mg-5Gd 48 h DCT sample displays nearly 50% less twins than the Mg-2Gd 48 h DCT counterpart. Additionally, with the Mg-5Gd cases, the twins display more segmented and smaller twins formation, secluded to individual grains, than in the Mg-2Gd case, which is clearly depicted by the high-resolution scans as given in Fig. 6. The examples of CHT and DCT sample in Fig. 6, also present the highly selective nature of the twins formation as well as the clear difference in the size and segmentation of the twins between the two states of the same alloy. The pole figure projections (presented in Fig. 4g and f) also show a stronger scattering of the orientations of the crystals that also directly impact the twins formation that show a much more scattered character (see particularly the $\{10\bar{1}2$ and $\{10\bar{1}3\}$ projections) that supports the claim of smaller and more segmented twinning, possibly also related to multiple twinning modes [43].

The Mg-10Gd samples show a stronger effect of the alloying, which predominantly shows a significantly lower twinning activation regardless of the sample state (see Fig. 5). Surprisingly, even after 48 h of DCT, the samples display minor formation of twins that does not increase significantly with application of DCT (although still 20% more than in the 48 h case against the 8 h one) and the twinning is limited to individual grains like for the Mg-5Gd (compare Fig. 5b with 53e). The pole figure projections confirm that twins are indeed, present but develop a pronounced activation with extended DCT time along the different subvariant orientations (compare Fig. 5f with 5g) indicating that DCT time still matters even in this alloying state. To visualize this profound difference, high-resolution EBSD was done on selected CHT and DCT

samples of the Mg-10Gd alloy, presented in Fig. 7. The examples show on one hand how non-existent is the twinning in most of the CHT grains (Fig. 7a-e) and on the other how in certain DCT grains the twinning can be considerable (Fig. 7f-j), although still much rarer to find than in the case of lower alloyed counterparts.

3.2. Surface micro-residual stresses

The XRD residual stress measurements revealed a clear dependence of surface micro-residual stress on both Gd concentration and cryogenic treatment duration (Fig. 8). In the CHT condition, all Mg-Gd samples exhibited compressive residual stress in all measured directions. At 0° , the compressive stress increased with Gd content, from -125 MPa in Mg-2Gd to -131 MPa in Mg-5Gd and -179 MPa in Mg-10Gd. This trend is consistent with the sum of the stress fields measured at 0° , 45° and 90° . The measurements are taken along the longitudinal and transverse directions and shear deformation. The increasing Gd concentration enhances solute supersaturation and promotes precipitation during the extrusion process and prior heat treatment that subsequently results in lattice distortions that generate internal strain and residual stress fields in the magnesium matrix [34]. The microstructural observations presented in Section 4.1 support this interpretation, particularly in relation to the presence of high distortion fields determined with KAM that are highest with lowest alloying content. The compositional sensitivity of residual stress in Mg-RE alloys has also been reported in studies examining cooling-rate-dependent stress evolution in Mg-Gd-based systems [44].

Following DCT, residual stress showed a strong dependence on the DCT exposure time. For all alloys, shorter DCT exposure of 8 h reduced the magnitude of compressive residual stress compared with the CHT state in all measured directions, dropping to the average range of about -40 MPa. This stress state is sustained after 24 h of DCT, except for the Mg-5Gd alloy that returned to a stronger compressive character already after 24 h of DCT. This is followed by the other alloys after 48 h, at which

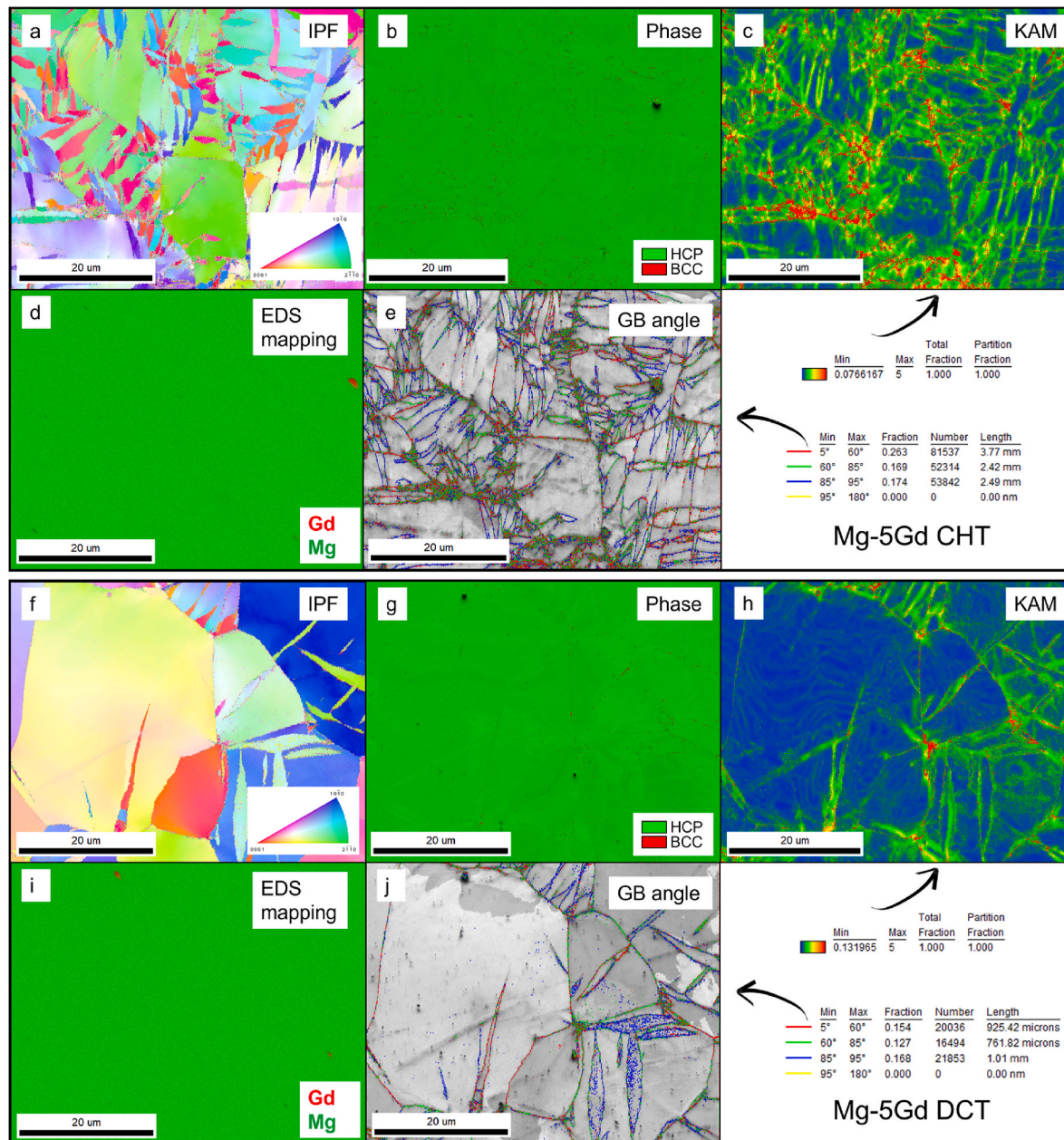


Fig. 6. Microstructural characterization of the Mg-5Gd alloy in the CHT and DCT-24 h conditions. (a-e) Characterization of the CHT condition: (a) inverse pole figure (IPF) map showing grain orientation distribution, (b) Phase identification map confirming HCP Mg as the dominant phase with Mg_xGd as a minor phase, (c) kernel average misorientation (KAM) map indicating local strain distribution, (d) EDS elemental mapping illustrating the spatial distribution of Mg and Gd, and (e) grain boundary misorientation angle distribution. (f-j) Characterization of the DCT-24 h condition: (f) IPF map, (g) phase identification map, (h) KAM map, (i) EDS elemental mapping, and (j) grain boundary misorientation angle distribution, showing the microstructural evolution.

all alloys exhibit on average about -120 MPa for the 0° and 45° component and -80 MPa for the 90° component. Similar stress-relaxation behaviour has been associated with microstructural rearrangement processes during DCT, including dislocation redistribution and precipitate modification in other magnesium alloy, albeit with additional 2-step aging treatment [45]. Nevertheless, this interpretation is consistent with this study's EBSD results showing increased grain subdivision and boundary reconfiguration without measurable changes in phase fraction, while showing consistency with the texture-related features that also impact the stress distribution.

The markedly faster change in the magnitude of the compressive stress for the Mg-5Gd alloy after 24 h of DCT indicates a distinct time-dependent response. However, this does give insight that similarly to other alloys system, such as ferrous alloys [32,46,47], DCT might have

peak efficiency at around 24 h of DCT. This would be consistent with the continuous observations and statements that DCT stimulates best changes in defects and solutes exchange at a particular timeline that with further excessive DCT exposure results in further modifications that can also negatively impact the final properties of the treated alloy. This has been particularly associated to solute redistribution [48] and their inherent binding with matrix [32,49]. This supports the potential explanation that the effectiveness of DCT depends on alloy composition, with an optimal holding duration observed at intermediate Gd content. These results suggest that the residual stress response in Mg-Gd arises from a combination of solid-solution related lattice strain, redistribution of defects and the activation of twinning nucleation and growth. The stronger compressive stress observed in Mg-5Gd after 24 h DCT, and thus also 48 h for all the alloys, therefore likely reflects a tipping point

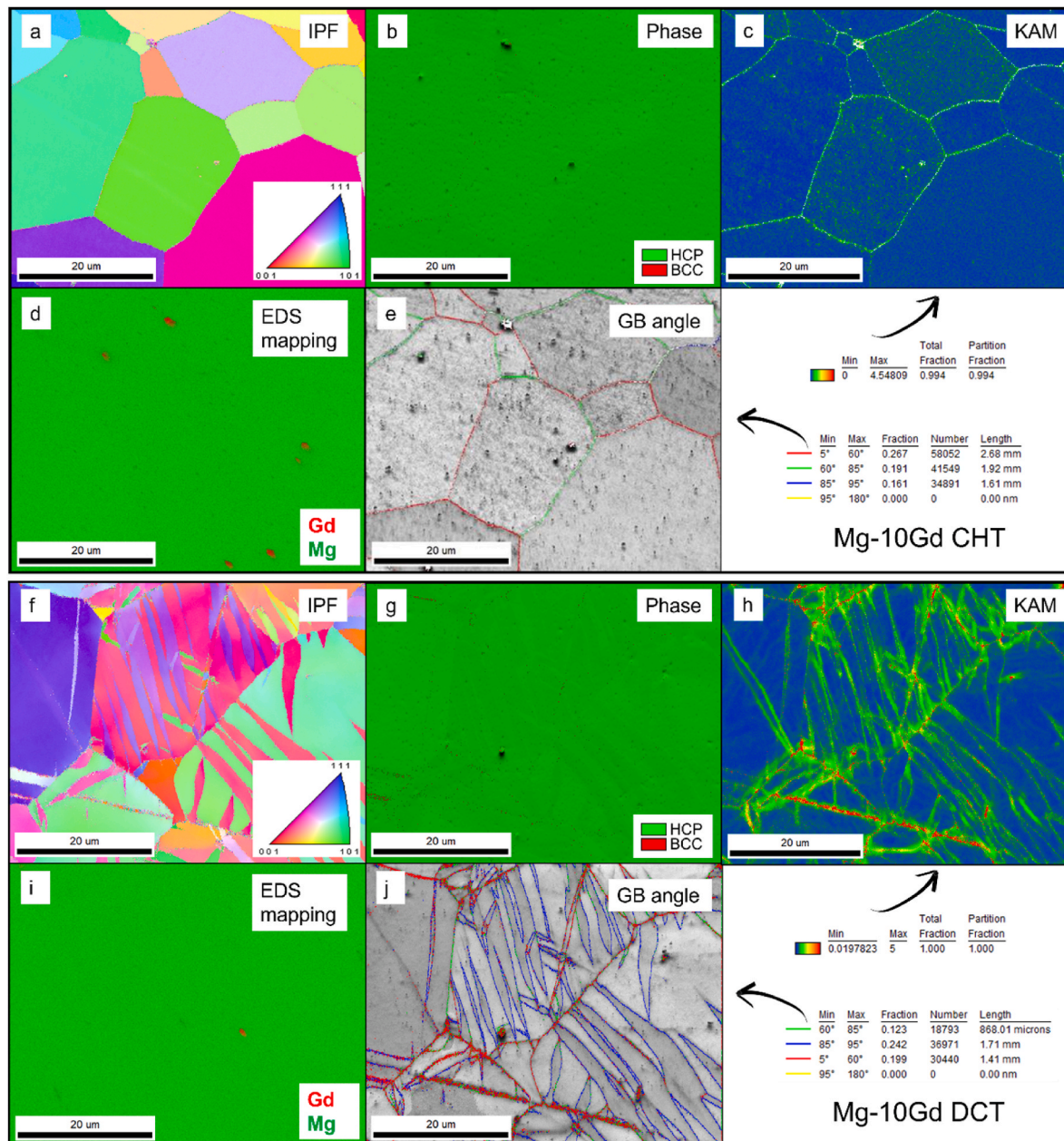


Fig. 7. Microstructural characterization of the Mg-10Gd alloy in the CHT and DCT-24 h conditions. (a-e) Characterization of the CHT condition: (a) inverse pole figure (IPF) map showing grain orientation distribution, (b) Phase identification map confirming HCP Mg as the dominant phase with Mg₂Gd as a minor phase, (c) kernel average misorientation (KAM) map indicating local strain distribution, (d) EDS elemental mapping illustrating the spatial distribution of Mg and Gd, and (e) grain boundary misorientation angle distribution. (f-j) Characterization of the DCT-24 h condition: (f) IPF map, (g) phase identification map, (h) KAM map, (i) EDS elemental mapping, and (j) grain boundary misorientation angle distribution, showing the microstructural evolution.

between these interrelated mechanisms, which appears less effective in the lower-Gd alloy and becomes saturated at higher Gd content.

3.3. Microhardness

The microhardness (Fig. 9) progression of Mg-Gd alloys under DCT exhibited a clear dependence on both alloy composition and treatment duration (Table 1). In the CHT state, Mg-2Gd displayed a hardness of 61 ± 3 HV, Mg-5Gd was softer at 46 ± 6 HV, and Mg-10Gd was the hardest at 83 ± 1 HV, consistent with the proposed effect of increasing Gd content as explained for the residual stress dependency. Following DCT, Mg-2Gd experienced an initial decrease in microhardness to 45 ± 1 HV after 8 h, with partial recovery to 57 ± 3 HV at 24 h and a subsequent decline to 48 ± 3 HV at 48 h. Mg-5Gd showed a pronounced increase in

microhardness, rising from 46 ± 5 HV in CHT to 62 ± 5 HV after 8 h and reaching a maximum of 80 ± 6 HV after 24 h, similar as seen for the Mg-2Gd case in relative perspective between the different DCT samples. The subsequent reduction to 60 ± 2 HV at 48 h confirms the time-dependent effect of cryogenic exposure as also postulated based on the residual stress measurements. The maximum hardness at 24 h can be rationalized by a combination of solute-induced lattice strain and the refinement of the microstructure through twinning which provide effective obstacles to dislocation motion. But the complete description and evolution of the interacting mechanisms will be discussed separately in more details in Section 3.4.

In contrast to Mg-2Gd and Mg-5Gd alloys, the Mg-10Gd alloy exhibits nearly constant hardness across all conditions, with values remaining at 83 ± 5 HV regardless of cryogenic treatment duration. At

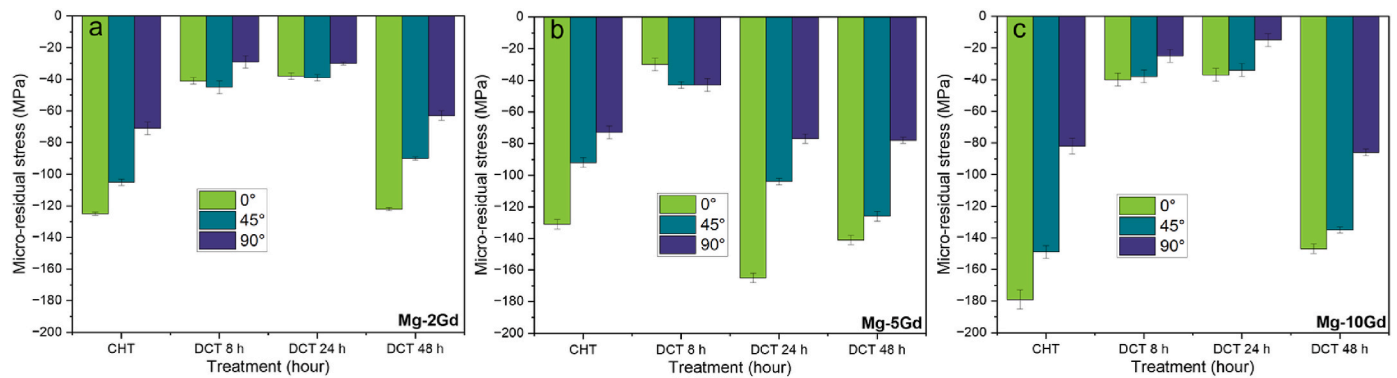


Fig. 8. Surface micro-residual stresses (MPa) measured by X-ray diffraction (XRD) for the investigated alloys: (a) Mg-2Gd, (b) Mg-5Gd, and (c) Mg-10Gd. All measured values are negative, indicating compressive residual stresses present within the microstructure of the alloys.

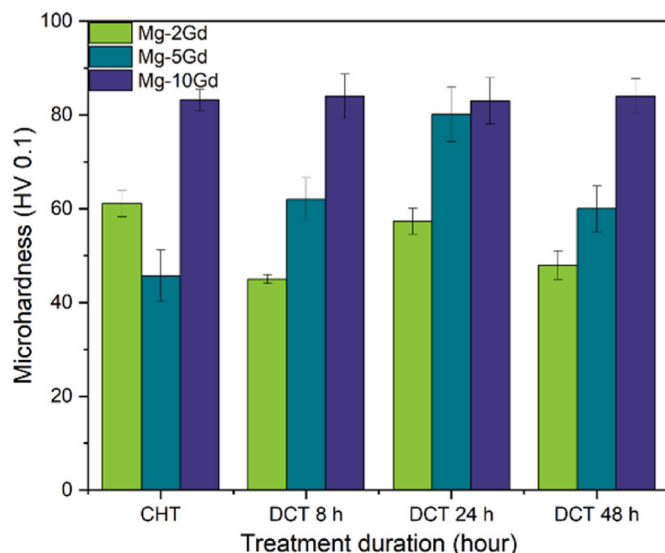


Fig. 9. Vickers microhardness (HV 0.1) of Mg-2Gd, Mg-5Gd, and Mg-10Gd alloys measured for CHT (extruded) and after DCT for 8 h, 24 h and 48 h.

this higher Gd concentration, solid-solution strengthening and precipitation hardening introduced during prior heat treatment and extrusion likely dominate the mechanical response, so the primary mechanisms of DCT, namely the redistribution of internal stresses and solute atoms, have a negligible effect on hardness and, consequently, on the alloy's strength. Overall, the microhardness trends demonstrate a composition-dependent response to DCT. Low-Gd alloys experience softening due to pronounced lattice-strain relaxation and limited solute strengthening, intermediate-Gd alloys exhibit significant hardening after 24 h of cryogenic treatment due to the synergistic interaction of multiple mechanisms, and high-Gd alloys remain largely unaffected because of pre-existing strong solid-solution and precipitation strengthening.

3.4. Insights into the underlying mechanisms

The combined experimental results indicate that the response of Mg–Gd alloys to DCT is governed primarily by cryogenic-induced lattice contraction interacting with pre-existing precipitate–matrix strain fields, rather than by phase transformation. However, DCT's modification of the stress fields is accompanied by the redistribution of dislocations, Gd solute atoms, and the reduced local energy landscapes, which in turn allows the activation of twinning planes that minimize the prior strain energy originating from the extrusion process. To this extent, we propose that the energy minimization is a result of multiple mechanisms

activated by DCT. The pronounced effect of twinning is a result of the incomplete dynamic recovery of the residual stresses present in CHT (extruded) material, which are relaxed through solute recombination, dislocation redistribution, and, consequently, enhanced twinning (see sketch of mechanism in Fig. 10). A key outcome is the direct reduction of compressive residual stresses following the application of DCT, as observed for all alloys regardless of the alloying content. This consistent effect confirms that the resulting defects and stress inhomogeneities are reformed with DCT, which also, in turn, reduce the hardness of the Mg–2Gd alloy. However, with prolonged DCT time, the stress state eventually returns to higher compressive stresses. This is explained by the activation and additional formation of twin structures, which result in higher stress fields due to occupancy of the twinning boundaries with Gd, as observed in such systems by Nie et al. [50]. The proposed stochastic activation of twins with DCT is also supported by literature on an AZ31 Mg alloy, which observed DCT-induced twinning with in-situ EBSD [51]. In turn, the twinning growth and increased formation indicate that sufficient energy, similar to a pre-straining effect [52,53], is present in the system to establish solute segregation not only at boundaries but also within the matrix itself.

Such mechanism enables the formation of nm-sized solute zones that contribute to the higher mismatch within the matrix, but also enables the homogeneous lock-in of the solute in the matrix from the random defect zones and excess-alloyed regions [54,55]. This is confirmed through APT probing of the CHT and DCT samples (see Fig. 10 and Fig. S2a–d) that confirms the higher Gd content of the matrix in the DCT case, while no pronounced larger-size precipitates are detectable. However, the APT does show statistically a higher amount of nm-sized pairs of Gd atoms that indicates not only a higher number of them with smaller size, but a significantly different distribution of the DCT case over the CHT one (see Fig. S2e). This would support the proposed theory that DCT through the introduction of shrinkage pressure and defect and alloying elements redistribution induces significant energy landscape modification that induce solute segregation formation similarly to the observed ones with aging [56] and dynamic precipitation. This mechanism also supports that the DCT-induced strengthening mechanism will not contribute to the strength and thus hardness of the Mg-10Gd alloy due to the prior precipitation hardening that is induced by the extrusion process and enabled by the high Gd content. And in the case of the Mg-2Gd case, the low alloying content combined with the removal of defects and redistribution of alloying elements reduces the overall strengthening of the microstructure, resulting in overall lower hardness of all DCT samples against the CHT one.

However, for the intermediate Mg-5Gd alloy, solute segregation strengthening and twinning act synergistically, resulting in a non-linear dependence of the material properties on DCT exposure time. This behaviour is particularly related to intermediate twinning propagation and widening, which are modified by the presence of solute

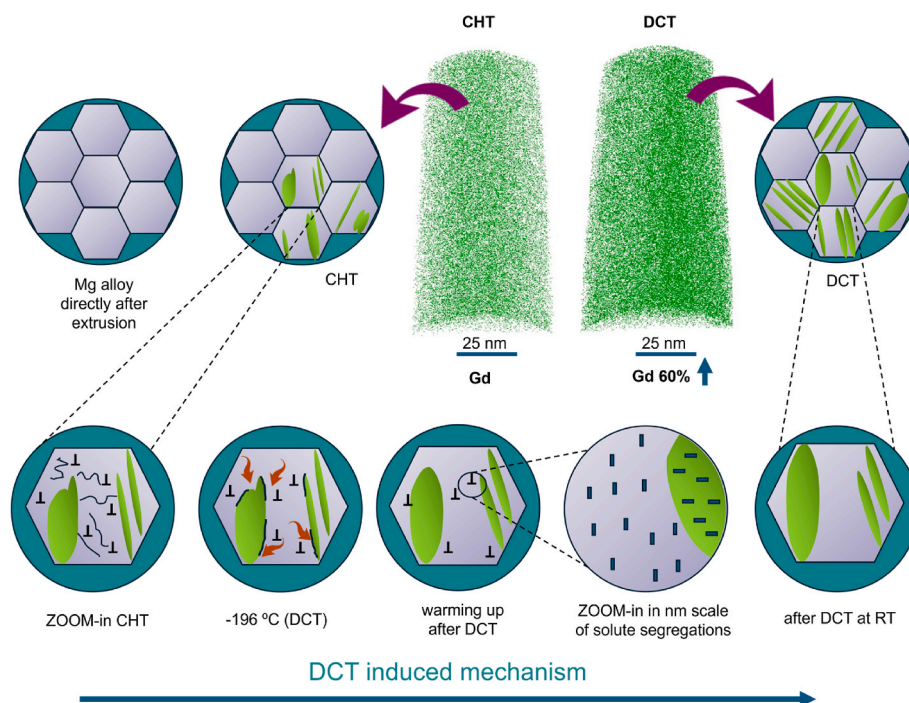


Fig. 10. Schematic representation of the DCT induced mechanism on the Mg–Gd alloys. The image also presents the APT data of the CHT (extruded) and 24 h DCT Mg–5Gd samples that depicts the considerable Gd enrichment with the application of DCT.

agglomerates and precipitates [57]. The effect of such a mechanism is reflected in the presence of segmented and sawtooth-shaped twins [58] which are a predominant feature within individual grains of the Mg–5Gd alloy (Fig. S3a–c). These atypical twins suggest that activation of additional basal and prismatic slip systems is possible in the material, likely promoted by changes in the stress state and the pinning of twinning boundaries by local precipitates, leading to the formation of twinning steps and a combined basal–prismatic interface [59]. These features have also been identified in the Mg–5Gd DCT samples through grain boundary analysis of the EBSD data (Fig. S3d). However, twinning alone explains only the general increase in hardness of Mg–5Gd with increasing DCT time and does not account for the decrease in hardness from 24 h to 48 h of DCT, a similar trend also observed for the Mg–2Gd alloy. The explanation therefore likely lies in recovery processes and solute segregation. After strong etching of the Mg–5Gd 24 h DCT sample, underlying polygonization becomes visible within individual large grains that exhibit limited dynamic recrystallization (DRX) (Fig. S3e–f). These polygonization features become visible due to the increased Gd content (Fig. S3f), which may promote partial Gd depletion and thereby limit the availability of solute for segregation strengthening as illustrated in Fig. 10. We therefore propose that increasing DCT time promotes a higher degree of polygonization, reaching a state similar to “peak aging” at approximately 24 h. Beyond this point, excessive polygonization reduces the subsequent strengthening effect due to insufficient Gd availability, leading to the formation of Gd-rich and Gd-poor regions near low-angle grain boundaries. This effect is particularly important when the lower alloying content results in a limited strengthening contribution from low-angle grain boundaries [60] compared with solute segregation strengthening.

4. Corrosion resistance and passivation layer at ambient conditions

The results presented here focus on Mg–5Gd, as this showed a positive trend with regard to DCT. Results for the other two evaluated Mg alloys (Mg–2Gd and Mg–10Gd) are presented in the supplementary material in Fig. S4–S7. The electrochemical behaviour of the Mg–5Gd alloy

under different treatment conditions was evaluated in 0.9 wt% NaCl solution, and the results are presented in Fig. 11a–c. The chloride-containing electrolyte provides an aggressive environment in which magnesium alloys undergo active dissolution accompanied by partial surface film formation and breakdown. As shown in Fig. 11a, the cyclic polarisation curves exhibit the characteristic anodic response of Mg in Cl media. Upon scanning from the OCP toward the anodic direction, the current density initially increases as a result of active magnesium dissolution. Concurrently, the dissolved Mg species react with hydroxide ions to form $\text{Mg}(\text{OH})_2$, leading to the precipitation of a surface film. This process is typically associated with a local increase in pH near the electrode interface. Although the resulting $\text{Mg}(\text{OH})_2$ layer can offer partial protection, it is thermodynamically unstable in the presence of chloride ions (Cl^-) and may transform into soluble MgCl_2 species [61]. The repeated formation and chloride-induced breakdown of the hydroxide film govern the corrosion behaviour of Mg alloys in NaCl solution, resulting in active dissolution throughout the forward anodic branch with no stable passive region. During the reverse scan from anodic to cathodic potentials, a reduction in current density is observed, indicating partial repassivation and surface-layer stabilization. This asymmetry is typical of magnesium corrosion: anodic polarisation thickens the film but renders it increasingly porous and mechanically unstable, while anodic hydrogen evolution within film pores contributes to disruption and prevents true passivation [62]. Repassivation is more pronounced under cathodic or near-equilibrium conditions, where local alkalization favours reformation of a more protective hydroxide film [61].

Differences in hysteresis loop areas (Fig. 11a) are evident across treatment conditions. The CHT sample exhibits the smallest loop, indicating the lowest polarisation resistance and highest uniform corrosion rate. The DCT-8 h and DCT-48 h samples display intermediate loop diameters, while the DCT-24 h sample shows the largest loop, reflecting the highest polarisation resistance and lowest uniform corrosion rate. These trends suggest that 24 h of cryogenic treatment enhances the protective properties of the surface film under near-equilibrium conditions, whereas 48 h primarily improves repassivation during cathodic polarisation.

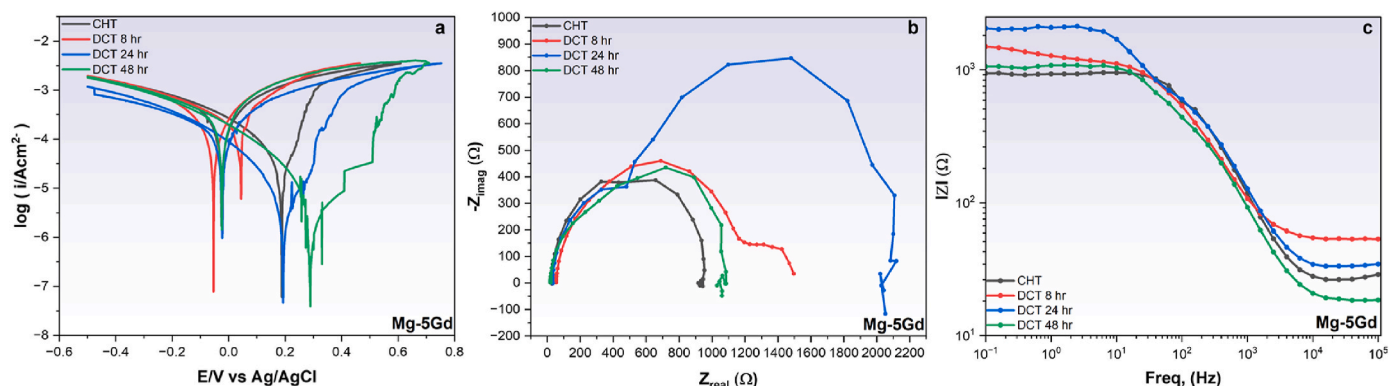


Fig. 11. Electrochemical corrosion behaviour of Mg-5Gd alloy in 0.9 wt% NaCl solution after CHT (extruded) and DCT with different soaking times: (a) CP curves for CHT and DCT-treated samples with soaking times of 8, 24, and 48 h, illustrating the anodic dissolution and repassivation behaviour; (b) Nyquist impedance spectra obtained at open circuit potential for the same conditions, showing variations in polarisation resistance and corrosion kinetics. (c) Bode magnitude plots illustrating the frequency-dependent impedance response.

The impedance response at OCP and Bode plot, Fig. 11b–c respectively, provide insight into the protective characteristics of the surface film under near-equilibrium conditions. The Nyquist plots exhibit a dominant high-frequency capacitive response, with a tendency toward a second, more depressed capacitive feature in the medium-frequency range. This second feature is not fully resolved into a distinct semi-circle but appears more noticeable for the DCT-24 h and DCT-8 h conditions, suggesting the presence of overlapping or partially resolved time constants. The low-frequency intercept (higher $|Z|$) reflects the overall resistance of the system, including contributions from the surface film and charge-transfer processes; higher impedance at low frequencies generally corresponds to greater resistance to corrosion, consistent with the slower corrosion rates [63].

Mechanistic studies on magnesium corrosion indicate that the high-frequency response in impedance spectra is primarily associated with the barrier properties of the thin inner surface film, mainly dense MgO. This response is typically modelled using a film resistance in parallel with a constant-phase element to account for non-ideal capacitive behaviour arising from surface roughness and heterogeneity. The diameter of the high-frequency semicircle reflects the resistance of the surface film and its protective ability, while the effective capacitance derived from the CPE provides an estimate of the film thickness [63]. The medium-frequency region, when discernible, is commonly linked to mass transport and ion diffusion through the more porous outer Mg(OH)₂ corrosion-product layer and to electrochemical reactions at the metal/film interface, including magnesium dissolution and localized interactions within film pores. This interpretation observed by Song and Unocic [62], who associated the intermediate capacitive feature with interfacial processes between the surface film and the underlying substrate. The often depressed and overlapping appearance of this medium-frequency feature is consistent with the heterogeneous, porous nature of the MgO/Mg(OH)₂ film, which frequently prevents the clear separation of these time constants in Nyquist spectra.

It should be noted that precise physical interpretation of individual time constants in magnesium EIS spectra remains an active area of research. Recent studies provide strong experimental support for the high-frequency loop originating from surface-film barrier properties [63], yet multi-time-constant behaviour in complex, porous films still requires further investigation. The present data are interpreted within this framework, while acknowledging that complementary studies, such as longer-term immersion in media of different pH, local electrochemical techniques, or advanced surface analysis would help refine mechanistic understanding specifically for Mg-5Gd.

The Raman spectra of the Mg-5Gd alloy (Fig. 12) provide insight into the chemical composition and structural characteristics of the corrosion product layers formed after electrochemical exposure. All samples

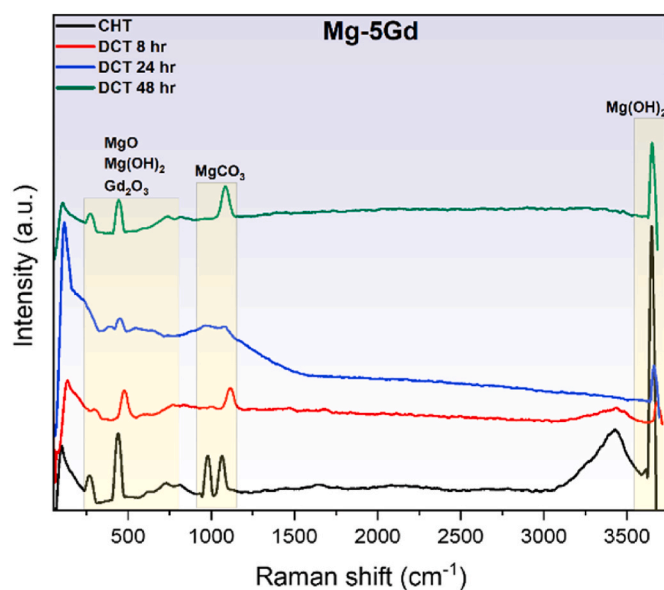


Fig. 12. Raman spectra of corrosion products formed on Mg-5Gd alloy after electrochemical exposure in 0.9 wt% NaCl solution for different treatment conditions: CHT, DCT 8, 24, and 48 h. The spectra show characteristic bands associated with Mg(OH)₂, MgO, Gd₂O₃, and carbonate species, with variations in peak width and intensity indicating differences in the crystallinity and structure of the corrosion product layer.

exhibit bands associated with MgO/Mg(OH)₂ in the region of approximately 270–450 cm^{−1}, corresponding to Mg–O lattice vibrations, together with a strong O–H stretching band near 3650 cm^{−1} characteristic of brucite-type Mg(OH)₂. Carbonate-related bands are also observed in the range of approximately 980 cm^{−1} and 1060–1090 cm^{−1}, which are commonly attributed to surface carbonation of Mg(OH)₂ during exposure to air after testing. A low-wavenumber band near 100–130 cm^{−1}, associated with Gd–Cl vibrations, is present in all spectra.

Comparison between the different treatment conditions reveals clear differences in band shape and width. The CHT sample exhibits the sharpest Raman features, indicating a relatively well-crystallized corrosion product layer. In contrast, the DCT-24 h condition shows noticeably broader bands, while the DCT-8 h and DCT-48 h spectra display intermediate behaviour with comparatively sharper features than DCT-24 h. Raman band broadening is generally associated with reduced crystallinity, smaller crystallite size, or increased structural

disorder in the corrosion products. The broader bands observed for DCT-24 h therefore suggest the formation of a less crystalline corrosion product layer.

These microstructural differences correlate with the electrochemical results. The DCT-24 h condition exhibits the highest polarisation resistance in the EIS measurements, indicating the lowest uniform corrosion rate. A less crystalline $\text{Mg}(\text{OH})_2$ layer may reduce ionic transport and chloride penetration compared with highly crystalline corrosion products, thereby enhancing its barrier properties. In contrast, the sharper Raman features observed for the CHT sample suggest a more crystalline corrosion layer, which may provide less effective protection, consistent with its lower polarisation resistance.

In addition to the Mg-based corrosion products, the Raman spectrum of the DCT-24 h sample reveals three weak but distinguishable bands at approximately 228 cm^{-1} , 383 cm^{-1} , and 563 cm^{-1} . These features are not observed in the CHT spectrum and are not clearly detectable in the DCT-8 h and DCT-48 h conditions. Although the intensity of these bands is relatively low, their positions are consistent with reported vibrational modes of Gd_2O_3 and may therefore indicate the presence of Gd-containing oxide species within the corrosion product layer after cryogenic treatment. The limited intensity and the absence of these bands in the other conditions suggest that such phases, if present, occur only in small amounts or are locally distributed. Nevertheless, their appearance in the DCT-24 h condition may indicate partial enrichment of Gd within the surface oxide/hydroxide layer, which could contribute to the differences in electrochemical response observed for this sample.

These observations are consistent with the microstructural evolution expected during cryogenic treatment. Prolonged exposure (DCT-48 h) may further reduce microstructural inhomogeneities, contributing to improved stability of the surface film that forms on polarising from anodic to cathodic potentials, as reflected by the larger hysteresis loop observed in the cyclic polarisation curves. Together, the electrochemical and spectroscopic results indicate that cryogenic treatment modifies both the microstructural state of the alloy and the characteristics of the corrosion product layer, thereby influencing the corrosion behaviour of the Mg-5Gd alloy.

Altogether, the combination of DCT-induced twinning, recovery and polygonization as well as solute segregation strengthening can explain the intricate yet well-defined effect of DCT on Mg-Gd alloys with different alloying content and their properties with DCT exposure times. With the established multi-mechanism explanation of the DCT effect, it is also proposed that with properly tailored aging after DCT, the microstructure could be further optimised to obtain high-strengthening of the alloy while also minimizing the recrystallization of the grains that would provide the best combination of strength and corrosion resistance. The resulting optimization could lead to similar observed effects of strengthening of alloys with aging treatment through precipitation hardening [58] and suppressed recrystallization normally achieved with the introduction of additional alloying elements [61] to enhance corrosion resistance.

5. Conclusions and outlook

This study demonstrates that DCT induces a strongly composition-dependent, non-linear microstructural response in extruded Mg-Gd alloys. While XRD analysis confirms that the α -Mg matrix remains dominant and that Mg_5Gd fractions remain largely unchanged after treatment, the results indicate that DCT primarily acts as a strain-mediated process that modifies defect structures, residual stresses, and solute distribution through lattice contraction under diffusion-limited conditions. The optimal processing condition identified was the Mg-5Gd alloy subjected to 24 h DCT. This treatment yielded the greatest improvement in microhardness and superior corrosion resistance in 0.9% NaCl, evidenced by the highest polarisation resistance and the formation of a less crystalline, more protective $\text{Mg}(\text{OH})_2$ surface layer containing minor Gd-oxide species, as revealed by Raman spectra. The

core mechanism responsible for these improvements involves cryogenic-induced lattice contraction that promotes dislocation redistribution, activation of tensile twinning, and nanoscale Gd clustering and solid redistribution within the matrix confirmed by APT. These synergistic effects enhance strengthening via twin boundaries and solute pinning while improving surface film protectiveness, reaching a peak efficiency at the intermediate 24 h holding time for this Gd content. In contrast, Mg-2Gd shows overall softening and Mg-10Gd remains largely unaffected due to dominant pre-existing solid-solution and precipitation strengthening. These findings establish DCT as a viable supplementary processing route for tailoring the performance of biodegradable Mg-Gd alloys, with the Mg-5Gd 24 h condition offering the most promising balance of mechanical integrity and controlled degradation for temporary implant applications. Further optimization combining DCT with controlled aging is expected to yield additional improvements.

CRedit authorship contribution statement

Saro Birgani: Methodology, Validation, Formal Analysis, Investigation, Writing-original draft, Writing-review & editing, Visualisation. Jad Abou-Saleh: Methodology, Validation, Writing-original draft, Writing-review & editing, Visualisation. Matic Jovičević-Klug: Methodology, Validation, Writing-original draft, Writing-review & editing, Visualisation. Björn Wiese: Methodology, Validation, Writing-original draft, Writing-review & editing, Resources. J. Manoj Prabhakar: Methodology, Writing-review & editing, Visualisation. Norbert Hort: Writing-review & editing, Resources, Supervision. Tim M. Schwarz: Methodology, Validation, Formal Analysis. Investigation, Writing-review & editing, Resources, Supervision. Michael Rohwerder: Writing-review & editing, Resources, Supervision. Patricia Jovičević-Klug: Conceptualization, Methodology, Validation, Formal Analysis. Investigation, Writing-review & editing, Visualisation, Supervision, Resources.

Declaration of ethics in publishing

Authors confirm that they have followed ethical guidelines published in Ethics Policy.

Declaration of generative AI use

Not applicable.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jmrt.2026.06.023>.

Data availability

The raw and processed data required to reproduce these findings are available from the corresponding author upon request.

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