Qualifying Electrically Conductive Cold Embedding-Media for Scanning Electron Microscopy

Hans-Bernward Besserer¹ · Viacheslav Boiarkin³ · Dmytro Rodman¹ · Florian Nürnberger^{1,2}

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Abstract The choice of embedding-media for metallographic specimen preparation, where subsequent scanning electron microscopy will be performed, is highly important since the quality of the analytical results in the specimen's near-surface region is limited by the properties of the embedding-material. Due to their high electrical conductivity and only slight tendency to form gaps between near the sample surface, warm curing embedding-media are usually best suited. Hot embedding-presses use relatively high pressures at elevated temperatures that can be detrimental for sensitive materials or coatings. Cold embedding with conventional embedding-media, which does not require high pressure mounting, requires compromising for a material with a lower electric conductivity, a higher amount of pores in the material, and inferior interfacial connectivity to the specimen. For this reason, the development of alternative electrically conductive and cold hardening embedding-media is of great interest for metallographic sample preparation of electron microscopy specimens. In the present study, various embedding-media were evaluated with respect to their suitability for scanning electron microscopy and a novel conductive mounting compound has been identified and qualified.

Keywords Cold embedding · Metallographic preparation · Scanning electron microscopy

Introduction

Scanning electron microscopy is a fundamental tool for the analysis of microstructure-property relationships. The choice of the embedding-media plays a decisively important role during metallographic preparation since the quality of analytical results can be influenced by the medium's properties. The quality of the embedding-media, i.e., its suitability for metallographic investigations, can be evaluated using seven criteria [1]:

- no reactions with the specimen material, the embedding mold, or the etchant,
- low viscosity during embedding,
- no pores or cavities after curing,
- little solidification shrinkage,
- adhesion to the sample with no gaps between surface and mounting compound,
- similar grinding and polishing behavior as the embedded material, and
- chemical resistance to the selected etchant.

Hot embedding-media are, as a rule, well suited for scanning electron microscopy because of their high electrical conductivity and marginal tendency to form gaps relative to cold mounting compounds. For example, the hot embedding-media ConduFast or PolyFast from Struers are frequently employed for SEM analyses since the high electrical conductivity, approximately $1.17 \times 10^2 \Omega^{-1} \text{ m}^{-1}$ in the case of ConduFast, minimizes the build-up of charge effects during SEM investigations [2, 3]. However, the use of a hot embedding-press with typical pressures of up to 25 MPa and

✉ Hans-Bernward Besserer
besserer@iw-uni-hannover.de

¹ Institut für Werkstoffkunde (Materials Science), Leibniz Universität Hannover, An der Universität 2, 30823 Garbsen, Germany

² ZFM – Centrum of Solid State Chemistry and New Materials, Leibniz Universität Hannover, Callinstr. 3a, Garbsen, 30167 Hanover, Germany

³ Department of Metal Forming, National Metallurgical Academy of Ukraine, Gagarina av. 4, Dnipropetrovsk 49600, Ukraine

temperatures in the range of 180 °C may damage or alter sensitive materials and may result in changes in microstructure [4]. Mechanical damages can occur on the one hand by embedding soft materials that deform plastically due to the compression in the mounting press, as exemplary reported for tin specimens [4]. On the other hand, cracks or spalling can occur at brittle specimen material. A case in point, e.g., thermally sprayed coatings were chipped due to the hot embedding, often has been noted [5].

An alternative method to avoid charging effects in SEM may be the microscopic examination by using environmental SEM (ESEM). Here, the microscope's sample chamber is evacuated only in the low vacuum range and a controlled volume of gas is introduced. The charge of the sample can partly be discharged over the atmosphere in the sample chamber. ESEM typically cannot achieve the same level of magnification as conventional SEM and the speed of image acquisition is greatly reduced. Thus, for some applications the ESEM is not a suitable alternative to the use of SEM in high vacuum mode [6].

Cold embedding-media are often used to eliminate the risk of artifacts due to temperature or pressure. Cold embedding-media are typically composed of acrylic, polyester, or epoxy resins. These materials differ in their cure time, polymerization temperature, chemical resistance, and shrinkage during curing, as summarized in Table 1 according to the specifications of leading manufacturers. These characteristics in particular are important in order to prevent formation of gaps between the sample and the embedding-compound [7].

Acrylic resins are often used because of their good chemical resistance, low shrinkage, and short curing times, which are typically less than 20 min. Likewise, polyester resins are highly resistant to chemicals, provide strong adhesion to the sample, and demonstrate low shrinkage as well. Epoxy resins are frequently employed as embedding-media because they feature the lowest shrinkage of cold embedding-media [4] and good adhesion, which helps avoid gaps. In addition, these resins exhibit also a high chemical resistance so that they will not cause problem during chemical etching [8]. Compared to acrylic and polyester, the epoxy resins require the longest time for curing, which makes on the one hand the embedding time-consuming, but on the other hand by the long duration in the liquid state a thorough degassing behavior is required.

Due to their low viscosity and vapor pressure, epoxy resins can be used for vacuum impregnation, for a better intrusion of the resin into porous substrate materials to minimize artifacts like pull-outs, cracks, or unopened porosity [7]. The main disadvantage of the cold embedding-media that are currently employed is that they have a significant lower electrical conductivity than hot embedding-media, because by cold embedding a lower density of the embedding-medium and thus of the conductive filler material in the medium is reached. Thus, electrical charge build-up due to the scanning electron microscopy can lead to image distortion. Additionally, because of shrinkage due to the curing, even if it is only slight, gaps can be formed between the specimen and the embedding-media, which is deleterious for near-surface analysis. Furthermore, pores in the embedding-compound formed due to the curing can potentially complicate the sample preparation and negatively affect the results of microscopic examination.

In the present study, various cold embedding-media were evaluated including two that are not currently employed for metallographic preparation. These embedding-media were evaluated based on the criteria: structure, electrical conductivity, adhesion to the sample, behavior during etching, and handling.

Materials and Experimental Procedure

Embedding-Media

For the present study, the epoxy resins CarboCond 171 and CarboCond 471 manufactured by Future Carbon GmbH, were selected as alternative resins for metallographic preparation. These materials are currently used only for floor coatings, cast components, and electrostatically discharging protective components [9]. These two epoxy resins are both containing about 0.6 wt% carbon nanoparticles and differ in their viscosity, which is 13 Pa s for CarboCond 171 and 3 Pa s for CarboCond 471. They are mixed with the hardener IPDX as intended by the manufacturer with a suggested hardener/resin mixing ratio of 100–32.

Since the IPDX hardeners contain plasticizers that can evaporate especially by curing under vacuum, an alternative hardener Epoxy1000 from Cloeren Technology GmbH with a mixing ratio of 5–1 was tested.

Table 1 Specific properties of resins used for cold mounting

Resin type	Curing time ^a	Characteristics	Polymerization temperature
Acrylic resins	5–20 min	thermoplastic	80–120 °C
Polyester resins	15–45 min	duroplastic	80–100 °C
Epoxy resins	360–720 min	duroplastic	55–75 °C

^a by curing at room temperature

For a comparative, the two commercially available, conducting, cold embedding-media Demotec 70 from Demotec Demel e.K. and EpoThin from Buehler Company were employed. These resins were each mixed using the specific hardeners and ratios suggested by the manufacturers (cf. Table 2). The EpoThin embedding-media, which is in the as-received state not electrical conductive, was mixed with nickel powder as conductive filler in the ratio 2–3.

Demotec 70 is a two-component material, comprising a powder component and a liquid component, on the basis of modified methyl methacrylate and conductive carbon.

Additionally, the hot embedding-media ConduFast from Struers GmbH was considered as a reference. ConduFast is an acrylic resin containing an iron filler. Here, the specimen was cured within 5 min at 25 MPa and 180 °C in the hot mounting press.

Substrate Material

Galvanized as the steel with a coating thickness of about 20 µm was used substrate, since the coating can be challenging for metallographic preparation. In particular, microstructural changes and coating damage can be caused by hot embedding due to the pressures and temperatures applied. The chemical composition of the steel specimens determined by glow discharge optical emission spectrometry is given in Table 3.

Curing Condition

To reduce the formation of pores due to the outgassing of the media during curing, three different curing methods were investigated. In addition to a curing at room temperature at ambient pressure (N), a curing in a weak vacuum at 0.05 MPa (V) and a curing in a pressure vessel at an elevated pressure of 0.15 MPa (D) were employed.

On the one hand, by curing under vacuum it can be expected that especially for the use with epoxy resins a better connectivity to the specimen can be reached by filling smallest pores in the specimen surface with

mounting material [4]. Otherwise the vacuum curing can result in unevenly distributed pores in the medium, because of rising gas bubbles from the liquid material during curing. On the other hand, it can be expected that curing at elevated pressure will not only reduce the formation of gas bubbles in the media but also improve the binding to the substrate by compressing the liquid embedding-material.

Evaluation

The procedure employed to assess the embedding-media properties is summarized in Fig. 1. The individual procedure steps have been assessed in regard to the execution as well as on the basis of the results. Some properties, such as the formation of pores or the electrical conductivity, were evaluated quantitatively. Yet, for the sake of simplicity, only the evaluation scheme, good (+), moderate (0), and insufficient (–), is used.

All embedding-media were prepared similarly. At first, the components were poured into a container in the specified mixing ratios (see Table 2) and stirred for at least 1.5 min. Next, the container with the embedding-compound was left to rest for 1 min so that the air bubbles, which formed during stirring, would exit. A light-curing polymer was used to fix the specimens during embedding and later completely ground away so as not to affect the SEM results.

Finally, the embedding-media were cured for comparison at room temperature, under vacuum or in a pressure vessel, as described in 2.3. Clearly, ease of handling is an important aspect as well, and thus, this was used as an additional criterion for evaluation.

Following mounting, automated grinding and polishing processes were carried out using the parameters given in Table 4. After preparation, pore density was determined from optical images recorded using a stereomicroscope.

The chemical resistance of the embedding-media to etchants is a key issue for microscopy analyses. Initially, a 2% nitric acid solution was employed as this represents a typical etchant. Subsequently, the effects on the embedding-media were analyzed using light optical microscopy.

Table 2 Mounting materials and mixing ratios analyzed in the present study

Trade name	Material	Hardener	Mixing ratio resin/hardener
Demotec 70	Methyl methacrylate and conductive carbon	Demotec 70	1:1
EpoThin	Epoxy resin, Ni-powder added	EpoThin	5:2
CarboCond 171	Epoxy resin with carbon particles	IPDX	100:32
		Epoxy1000	5:1
CarboCond 471	Epoxy resin with carbon particles	IPDX	100:32
		Epoxy1000	5:1
ConduFast	Acryl with FE particles	...	...

Table 3 Chemical composition (in wt%) of the galvanized steel

C	Si	Mn	P	S	Cr	Ni	Mo	Al	Fe
0.032	0.002	0.236	0.014	0.006	0.025	0.066	0.01	0.045	Balance

Fig. 1 Methodology and assessment criteria **Table 4** Parameters used for grinding and polishing

Processing step	Grain size or polishing finish	Paper/cloth	Force	Time	Cooling
1	220	SiC	20 N	2–7 min	Water
2	500	SiC	20 N	2 min	Water
3	1200	SiC	20 N	2 min	Water
4	2500	SiC	20 N	2 min	Water
5	6 μm	moderate-piled artificial silk	20 N	4 min	Ethanol-based lubricant
6	3 μm	moderate-piled artificial silk	20 N	2 min	Ethanol-based lubricant
7	1 μm	short-piled artificial silk	20 N	1 min	Ethanol-based lubricant

If no changes were observed after etching with the nitric acid solution, the procedure was repeated with the more aggressive Kroll's etchant, which contains nitric acid and hydrofluoric acid and is commonly used for aluminum and titanium alloys.

In order to measure the electrical conductivity of the embedding-compounds, these were analyzed without an embedded steel-sheet specimen. The conductivity was determined using an induction–capacity–resistance measuring device at three measuring points. The level of electrical conductivity thus determined is considered an

appropriate criterion to assess the suitability of an embedding-media as charging is a key problem for high-quality SEM analysis.

An reflecting light microscope was employed to quantify the size of gaps between the specimen and the embedding-media. A gap between the specimen and the embedding-media particularly impairs investigations of the specimen's edge region using SEM. Prior to the light microscopy investigations, the specimens were ground and polished but not etched. At first, the specimens were examined at 20 times magnification in order to obtain an

overall impression of the gap. The specimens were then investigated using a higher magnification ($\times 50$ or $\times 100$). Based on these images, the gaps occurring at the specimens' surface were measured. Once the gaps were measured, the next experiment was to determine how much a secondary electron image of the embedding-compound could be magnified without significant image distortion in a conventional (evacuated in the range of 280–480 Pa) SEM. Basically, the maximum, non-distorted adjustable magnification in this case is limited by the charge build-up.

Results and Discussion

Embedding

In the first stage of embedding, handling was evaluated. In accordance with the manufacturer's instructions, the mounting materials were all mixed, stirred, and were let to rest at room atmosphere for degassing prior pouring. Embedding-compounds that flowed sufficiently, so that complete encapsulation around the specimen is guaranteed, were evaluated as good. In this case, fewer disruptive air bubbles appeared during pouring. Thus, the embedding-compound's electrical conductivity is less impaired and adhesion between the specimen and the compound is improved. Table 5 summarizes the results regarding the embedding behavior of the commercially available, conducting, cold embedding-media. Both Demotec 70 and EpoThin are easy to handle, and the consistency permitted its uniform dispersion around the specimen. The curing times were, however, substantially different. Demotec 70 cured within 18 min, whereas EpoThin required up to 720 min.

Table 6 summarizes the results obtained for the epoxy-based resins CarboCond 171 and CarboCond 471 that were tested in combination with the two hardeners CarboCond IPDX and Epoxy1000. In all considered cases, the resulting embedding-compound showed a low viscosity and it was possible to produce a homogeneous dispersion in the specimen mold. The curing time for CarboCond 171 and CarboCond 471 were in the range from 360–480 min.

In order to evaluate the fraction of pores in the compounds, stereomicroscopy images of the polished surfaces were recorded. Three categories of pore densities were

used and examples are depicted in Fig. 2. As highlighted by rectangles in Fig. 2, the fraction of pores was determined using a region of interest (ROI) of 100 mm^2 .

In Fig. 3, the fraction of pores is given for the various embedding-compounds and different curing conditions. For the use in the SEM, the pore fraction in the material should be minimized. Since the existence of a few large pores can clearly affect the suitability for microscopy more than a larger amount of small pores, the average pore size is shown in Fig. 4 for the various embedding-media and curing conditions.

The embedding-media CarboCond 171 and 471 behave similarly and the pore density depends very much on the hardener and the curing conditions used. Comparing the pore fraction and the mean pore size (Figs. 3 and 4), it can be found that an increased outgassing during curing occurs mainly in combination with the hardener Epoxy1000. Thereby more large pores are formed and an increased fraction of pores in the material can be detected. The latter is particularly evident for curing at ambient pressure and room temperature.

Furthermore, comparatively strong outgassing occurs when curing EpoThin with Ni-powder. Here, smaller pores tend to form than by the combination of CarboCond embedding-media with Epoxy1000 hardener, whereas a large amount of these smaller pores results in a high pore fraction for the EpoThin embedding-medium with Ni-powder incorporated.

The embedding-medium Demotec 70 showed for all curing conditions good to moderate regarding the fraction of pores. Curing under vacuum resulted in larger pores, whereas by curing in the pressure vessel almost no pores could be observed.

Very promising is the combination of a CarboCond Embedding-medium with the manufacturer's recommended IPDX hardener as the average pore sizes are quite low and especially by curing at elevated pressure a small fraction of pores could be generated. Since the pore distribution around an embedded sample influences the electrical conductivity, different curing conditions for CarboCond 171/IPDX samples were prepared as depicted in Fig. 5, additionally.

The cross-section pictures reveal that the resulting pore distribution is best for the specimen prepared in the pressure vessel. Curing at room temperature produces

Table 5 Embedding behavior of conventional, conducting cold embedding-media

	Demotec 70 (1:1)	EpoThin with Nickel powder (2:3)
Embedding	very liquid compound, good dispersion around the specimen	liquid compound, good dispersion around the specimen
Evaluation	+	+

Good (+) moderate (0) insufficient (–)

Table 6 Embedding behavior of CarboCond 171 and CarboCond 471

	CarboCond 171 IPDX (100:32)	CarboCond171 Epoxy1000 (5:1)	CarboCond 471 IPDX (100:32)	CarboCond 471 Epoxy1000 (5:1)
Embedding	Liquid compound, good dispersion around the specimen	Liquid compound, good dispersion around the specimen	Liquid compound, good dispersion around the specimen	Liquid compound, good dispersion around the specimen
Evaluation	+	+	+	+

good (+) moderate (0) insufficient (–)

Fig. 2 Examples for the three categories of pore fractions used**Fig. 3** Fraction of pores for the various embedding-compounds and different curing conditions

mainly pores in the middle region of the sample, since these cannot degas via the surface of the sample during curing. In contrast, curing under vacuum leads to an uneven distribution of larger pores. Therefore, for both the resulting pore fraction, as well as the size of the pores, for this combination curing under vacuum is not to recommend. Thus, successful curing at elevated pressures in the pressure vessel for the CarboCond 171 embedding-medium resulting in a small pore fraction and small pore sizes.

Suitability for Grinding and Polishing, Chemical Resistance, and Electrical Conductivity

Despite the differences in the pore fraction in different materials, all of the prepared specimens were suitable for the procedure described in Table 6 regarding grinding and polishing without any restrictions or specific arrangements. Additionally, the Shore D hardness has been determined for the investigated mounting materials. As depicted in Fig. 6 for the CarboCond Materials, a higher hardness

Fig. 4 Average pore size for the various embedding-compounds and different curing conditions

Fig. 5 Sections of samples of CarboCond 171 with IPDX hardener cured at different curing conditions

(between 78 Shore D and 81 Shore D) compared to the other cold embedding-materials could be determined.

During the chemical etching tests, none of the investigated embedding-compounds exhibited structural changes. No visible changes of the embedding-materials were observed using a light microscope for etching with 2% nitric acid solution or Kroll's etchant.

To determine the electrical conductivity, samples of the embedding-media were prepared without embedding the substrate material. All the specimens were cured in the pressure vessel since here only the electrical conductivity was of interest.

Figure 7 shows the results of the measured electrical conductivity of the investigated cold embedding-media. The highest electrical conductivity was achieved with CarboCond 171 and CarboCond 471 in combination with the Epoxy1000 hardener. This was attributed to the carbon nanoparticles and the graphite mixed into the epoxy resin [8]. In comparison, the determined electrical conductivity of the well-established cold embedding-media Demotec 70 and EpoThin was $0.3 \times 10^{-3} \Omega^{-1} \text{ m}^{-1}$, which is significantly below those of the CarboCond cold

embedding-media in combination with Epoxy1000 hardener. All cold curing embedding-media featured a significant lower electrical conductivity in comparison to a specimen prepared using the hot curing embedding-medium ConduFast with a specific conductivity of $1.17 \times 10^2 \Omega^{-1} \text{ m}^{-1}$. Thus, all considered cold curing embedding-media show an electrical conductivity which is by several orders of magnitude lower than those of established hot curing embedding-compounds. As described, the density of the mounting material is decisive for the density of the conductive filler in the medium and hence the electrical conductivity. The density of the investigated warm curing embedding-medium ConduFast could be determined to 2.3 g/cm^3 . By contrast, the cold mounting materials Demotec 70 with 1.4 g/cm^3 and EpoThin with 1.7 g/cm^3 possess a significant smaller density. The density of the CarboCond embedding-media is for all resin-hardener combinations at about 1.2 g/cm^3 . That CarboCond mounting materials exhibit a higher conductivity even though they have the lowest density indicates a good function of the carbon nanoparticles as filler.

Fig. 6 Hardness of the investigated cold embedding-media

Fig. 7 Electrical conductivity of the investigated cold embedding-media

Fig. 8 Light microscopy micrographs of the region between the embedding-compound and the galvanized specimen with good (*left*; no gap), moderate (*middle*; 0.5 μm gap), and poor adhesion (*right*; 2 μm gap)

Light and Scanning Electron Microscopy Analysis

The connection between the embedding-compounds and the galvanized specimens after polishing observed by light microscopy is depicted in Fig. 8. The micrograph on the

right shows an embedding-medium with low adhesion to the specimen. In fact, a continuous gap between the embedding-medium and the galvanized coating is visible. A gap reduced in distance can be seen in the micrograph in the center, whereas the left micrograph depicts a specimen

Table 7 Evaluation of edge gaps between specimen and embedding-media

	Demotec 70			EpoThin Ni-powder			CarboCond 171 Epoxy1000			CarboCond 171 IPDX			CarboCond 471 Epoxy1000			CarboCond 471 IPDX		
Curing condition	N	V	D	N	V	D	N	V	D	N	V	D	N	V	D	N	V	D
Evaluation	–	–	–	+	–	+	+	+	+	+	+	0	0	+	+	0	+	+

0–1 μm = good (+); 1–2 μm = moderate (0); >2 μm = increased gap size (–)

Fig. 9 Secondary electron image of a charged specimen embedded in Epothin with Ni-powder (*left*) and a weakly charged specimen embedded in CarboCond 171/IPDX (*right*)

featuring a bonding between the specimen and embedding-compound but no gap. The light microscopy analysis of the interface revealed that the majority of the embedding-media exhibited good bonding to the specimen. Specimens possessing a gap greater than 2 μm were considered as inadequate for high-resolution SEM work.

The detailed gap analysis revealed that, with the exception of Demotec 70 and EpoThin with nickel powder cured in vacuum, all the remaining embedding-media including the CarboCond media exhibited either only small or no gap (see Table 7).

By random analysis of the charge strength in SEM, it could be determined that with the exception of EpoThin with Ni-Powder all embedding-media showed very little or no charge effects. This is exemplarily shown in Fig. 9, visualizing charge effects on an EpoThin sample and a charge-free image of CarboCond 171. However, in most cases these artifacts were observed locally only and with some distance to the interface so that these effects were negligible regarding imaging.

Conclusions

In the present study, various cold embedding-compounds were analyzed with respect to their suitability for high-quality SEM analyses. The embedding-compounds' behavior during preparation (handling, grinding, polishing, and etching), the quality of the resulting structure of the media (formation of gaps and pores), the electrical conductivity, and the quality of the SEM analyses (secondary electron imaging) were used as evaluation criteria. Two commercially available, conducting, cold embedding-media, which served as references and two

novel electrically conductive epoxy resins (CarboCond 171 and CarboCond 471) were studied. The electrically conductive epoxy resin, which had hitherto not been used for metallographic purposes, showed at least equivalent, and to some extent, better properties compared to the conventional, conducting, cold embedding-media.

The investigated cold embedding-media differ regarding viscosity, degassing behavior during curing, and the required curing time. Thus, the chosen curing conditions influence the formation of pores in the media. The best results were obtained by curing at an elevated pressure of 0.15 Mpa in a pressure vessel. In comparison to the established embedding-media Demotec 70 and EpoThin, the novel CarboCond media showed less but slightly bigger pores and an increased electrical conductivity. Despite low electrical conductivity compared to the hot mounting resin (ConduFast), these embedding-media featured only low charge effects in the SEM.

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