EPMA European Hard Materials Group

Ultrasonic Fatigue Testing of Hardmetals in the gigacycle regime Project
UFTH Stage 2a:

Extension
and
Ultrasonic three point bending system for fatigue studies of HM (3PB)

Consortium Agreement

Issued 30 October 2013

The Project – "UFTH Stage 2a" as defined in Annex 1
The Contractors –

- Institute of Chemical Technologies and Analytics, TU Wien, and Faculty of Physics, Nanomaterials group, Univ. Wien, Getreidemarkt 9/164, A 1060 Vienna, Austria: **TUW-UW**,
- CEIT Centro de Estudios e Investigaciones Técnicas de Gipuzkoa, Materials Department, Manuel Lardizabal Ibilbidea, 15 20008 Donostia-San Sebastián, Guipúzcoa, Spain: **CEIT**
- Material Division, NPL Management Ltd, Hampton Road, Teddington, Middlesex, TW11 0LW, UK: **NPL**
- Universitat Politècnica de Catalunya (UPC), C./ Jordi Girona 31, 08034 Barcelona, through CIEFMA (Centro de Integridad Estructural y Fiabilidad de Materiales, TECNIO member): **CIEFMA UPC**

The Coordinator –

- The European Powder Metallurgy Association, Talbot House, 2nd Floor , Market St., Shrewsbury SY1 1LG, England: **EPMA**

The Members - **paid up corporate EPMA members*** funding the Project

The Participants – The Contractors and the Members

UV = unanimous vote of Members and Contractors; MV = majority vote of 2/3 members or higher

Heads

1. The Members and Contractors agree to cooperate in order to complete the Project according to Annex 1.

2. All information generated under the Project will remain confidential to the Members during the Project and for FIVE (5) years after delivery of the final written report to

Members, and may only be disclosed to third parties (e.g. for dissemination purpose in PM Congress) with UV.

3. The Contractors agree to not carry out a similar project on hardmetals with organisations other than the Members until the completion of the project (delivery of the final report). The aforementioned obligation shall not apply to other entities of TUW other than to its performing entity Institute of Chemical Technologies and Analytics, to other entities of UW other than its performing entity Faculty of Physics, Nanomaterials group, and to other entities of UPC other than its performing entity CIEFMA research group.

   The Member is aware of the Contractors status as scientific research establishment, and, consequently, a generous attitude to publication shall be maintained. In order to ensure that no risk to potential patents is taken, however, no publication of any result from the Project shall be made without the Members written consent, which will not be unreasonably withheld. A Contractor shall supply the Members with the manuscript of the intended publication, and, within four (4) weeks, the Members will respond with permission, or otherwise, to publish the manuscript either as is, or after suggested changes in content have been made. Failure of a Member to respond as specified will be considered as consent by default.

4. The Members agree **to share equally the cost of the Project** (EUR 40,852 – EUR 6,000 - See Item 6) through a Project Fee of maximum **EUR 8,713** per Member according to the Payment Schedule detailed in 9 if Item 6 is agreed. If not the total cost of the project to be shared will be EUR 40,852. The required minimum number of Members is **four** unless the Members agree to exceed the maximum Project Fee.

5. **VAT**: The Project Fee is excluding VAT if applicable. Non UK participants do not pay VAT provided they give their VAT number to the EPMA. UK participants have to pay VAT regardless and then reclaim it.

6. **Special Funding** of the Project: By signing the Consortium Agreement the Company HILTI CORPORATION, 9494 Schaan, Liechtenstein agrees to fund the project with an additional budget of EUR 6,000 in addition to the Project Fee described in 4.

7. **UTFH results access premium**. New Members, who did not participate in all the UFTH Stage 1, UFTH Stage 1 Extension and UFTH Stage 2 projects, may participate in the UFTH Stage 2a project by settling a “UTFH results access premium” of **EUR 4,000** at the start of the UFTH Stage 2a Project. The premium will be used to decrease the Project Fee for the UFTH Stage 2 Consortium Members.

8. The Members also undertake to provide the Contractors with the necessary test specimens and their appropriate surface preparations (Work Package 0 “WP 0”). If no agreement on in-kind contribution between the industrial partners can be found, the EPMA will coordinate the WP 0 and charge equally each Member to cover the cost of WP0 plus an administrative fee of 10%.

9. **Payment Schedule**:
   For the Work Package 0 “WP 0”: Full payment within one month after invoice if necessary.
   For each other Work Package:
   - **50% at the start**,

- **50% after completion** of the Work Package and delivery of the Work Package report.

10. New paying members may be admitted during the Project by UV on payment of full Project Fee plus the “UTFH results access premium” and an additional reasonable premium (10%). The premium will be used to decrease the Project Fee for the UFTH Stage 2 Consortium Members.

11. Except for the deliverables of the Annex 1, each Participant will retain the Intellectual Property for any other outcomes of the project.

12. Except for the term 4, all the terms of this agreement may be changed by UV.

Coordination will be by the EPMA, who will have responsibility for invoicing, day to day liaison with the Contractors and keeping Members informed. The EPMA will operate under the same confidentiality agreement as Members and the EPMA President will be arbitrator for unresolved disputes.

Signatures: signed individually by all Members and Contractors

ORGANISATION:

NAME:

(Date signed)

<u>*If you are not an EPMA member please contact Dr Olivier Coube, EPMA Technical Director, oc@epma.com</u>

Annex 1:

Work Packages in the Ultrasonic Fatigue Testing of Hardmetals in the gigacycle regime Project

Part 1: Stage 2a-Extension

The principle is to use the remaining samples from the UFTH 2 stage for further characterizations. The idea is to carry out simplified S-N curves with 3 levels of Stress Amplitudes (High, Medium and Low) and reduced sample numbers. Three specimens each will be tested at three different stress levels, i.e. nine specimens per grade. Two grades will be further studied: E10 and SHM-C. Surface finishing: The samples will be polished to check the surface defects effects on the S-N curves. The annealing condition should remain the same (800°C at 1hr in vacuum).

WP 0 Selection and Surface Preparation of Test Specimens

Solutions will have to be found regarding the polishing steps, scanning electron microscope (SEM) studies or annealing steps according to Item 8 of the Consortium Agreement.

Distribution of tasks: Industrial Partners

- Polishing of between 18 and 25 samples (to be defined) : Industrial Partner or Alternative
- Scanning Electron Microscope (SEM) studies of max. 25 samples (to be defined): Industrial Partner or Alternative
- Annealing of between 18 and 25 samples : Industrial Partner or Alternative

Cost:
- Contribution by Members (industrial partners): cost not included here. If no agreement on in-kind contribution between the industrial partners can be found, the EPMA will coordinate the WP 0 and charge equally each Member to cover the cost of WP 0 plus an administrative fee of 10% according to Item 8 of the Consortium Agreement.

Estimated duration: ca. 2-3 Months for WP0.
Deliverables: Polished Test Specimens, SEM characterization

WP 2a-Extension: Extension of the UFTH Stage 2-MRE WP (Microstructure-Related Effects "MRE")

Program:
Measurement of S-N curves – E10 and SHM-C grades, Surface preparation: Ground Annealed and Polished, check of Residual Stresses:

Distribution of tasks:

- Supply of material data: **Industrial Partners**
- Surface preparation: **See WP0**
- Measurement of residual stresses in 2 different grades of 2 samples after the polishing step: **CEIT**
- Measurement of residual stresses in 2 different grades of 2 samples after the annealing step: **CEIT**
- Gigacycle fatigue testing -> 2 simplified S-N curves with 3 levels of Stress Amplitudes (High, Medium and Low) and reduced sample numbers: **TUW/UW**
- Consulting regarding the damage evolution and crack/microstructure interaction (if subcritical fatigue crack growth is discerned) Fatigue mechanics analysis and detailed view (in one or two samples) of well-defined failure origins: **UPC**
- Consulting regarding Fractographic and structural evaluation: **NPL,**

Cost of WP2a-Extension:

- Contribution by industrial partners: cost not included here (WP 0)
- TUW/UW: 8,000 €
- NPL: 1,000 €
- UPC: 1,000 €
- CEIT: 1,000 €
- EPMA Mngt fee: 1,100 €

Total Cost of WP 2a-Extension = 12,100 €

Estimated duration of WP 2a-Extension: ca. 10 Months in total

ca. 4 Months from samples delivery

Deliverables (TUW/UW, CEIT, NPL,UPC):

- Intermediate reports and presentations
- Final reports
- Measurement data points (xls file)

Part 2: Stage 2a-3PB

An ultrasonic three point bending system for fatigue studies of HM

Adaptation of testing equipment

Introduction

Ultrasonic resonance fatigue testing of materials has been available since the late 1950s and has been successfully introduced for PM materials in the 1970s, at that time mainly for fully dense PM lightweight (Al base, Ti base) materials and refractory metals and subsequently also for sintered steels. Its main benefits are as follows:

- It enables testing up to the gigacycle range within reasonably short times, for 10E10 cycles about 5 days being required at standard 20 kHz frequency (compared to about 8 years for a servohydraulic testing machine). This enables studying the fatigue behavior of materials at very high loading cycle numbers, in particular with regard to the question if a “fatigue limit” exists or not; so far the results indicate that it does not exist for any real material. This is of crucial importance for components loaded to high but poorly defined N such as engine components or railway axles.
- Gigacycle testing is also an excellent tool for identifying singular defects in materials since in particular for high strength, high purity materials the fatigue endurance strength is defined by the largest defect in the loaded volume (inclusion, pore, pore cluster). In that respect, for gigacycle fatigue loading metallic materials show a very similar behavior as ceramic materials in static loading. In particular for ultra-high purity steels, such as spring or bearing steels, gigacycle resonance testing is the only feasible way to detect (and analyze) the last remaining inclusions in the materials since the inclusions are too small for nondestructive methods and too rare for metallographic investigations.

So far, gigacycle testing has been done in push-pull mode using dumbbell shaped specimens. In this case, the nominally loaded volume is much larger than in case e.g. of bending loads, and thus also the chance of identifying strength-limiting defects is higher. For standard metallic materials such as sintered steels, tool steels, Ti and Al alloys, manufacturing of the specimens is not too difficult, not even preparing the thread that has to be machined at one end to fix the specimen to the resonating system. For high strength steels, machining is usually done in the soft annealed state, with subsequent heat treatment. Things are however much more difficult for materials that are intrinsically hard such as hardmetals, ceramics or composites; here; machining in the final state is at best extremely expensive, at worst virtually impossible. For hardmetals, the solution so far has been to do machining of presintered bars, then sintering, grinding and stress relieving anneal, which makes specimen preparation a laborious and very expensive task which also contains some uncertainties regarding the precision of the thread machining of which has to be done such as to compensate for the sintering shrinkage.

Therefore, modifying the testing equipment to enable ultrasonic fatigue testing in bending load was regarded to be a highly attractive alternative that enables using specimens with much simpler geometry, typically rectangular bars. Here it must be considered that bending loads result in a much lower nominally loaded volume that push-pull loading, and therefore bending tests will yield reliable results only for such materials in which the strength is microstructure controlled, as opposed to defect-controlled as typical for ceramics. The studies carried out in UFTH projects Stage1 and 2 have however clearly indicated that for hardmetal it is the microstructure that defines the endurance strength and not singular defects; therefore it can be assumed that the ultrasonic bend testing will result in similar results as push-pull testing, but with much less effort and expenditure regarding specimen manufacturing. Of course, this hypothesis will have to be verified by parallel testing.

Basic principle of an ultrasonic three-point bending system

The basic idea is to transform the longitudinal vibrations into transversal vibrations of a resonating specimen. The longitudinal resonating vibrating system is coupled at the position of maximum displacement to the specimen, which is tuned to resonance, see fig. 1. The entire system consists of an excitation system and a resonating HM-sample. The length of the sample is a wavelength and can be tested in combination with a 3-point bending device. The used frequencies are in the range of 20 kHz, which means that as stated above, one can obtain failure in a few hours compared to the standard bending fatigue test methods which are in the order of days and month.

Fatigue loading of the HM samples, which may *be simple cylindrical rods or samples with a rectangular cross-section*, with sufficient high amplitudes may lead to crack initiation and crack propagation and final failure. Thus it is feasible to determine fatigue lifetime curves and fracture mechanics data in short testing times. In this case, also static loads can be superimposed to the dynamic loading (i.e. testing at R ≠ -1 is possible, in contrast to push-pull testing). Crack initiation and propagation can be measured using optical systems.

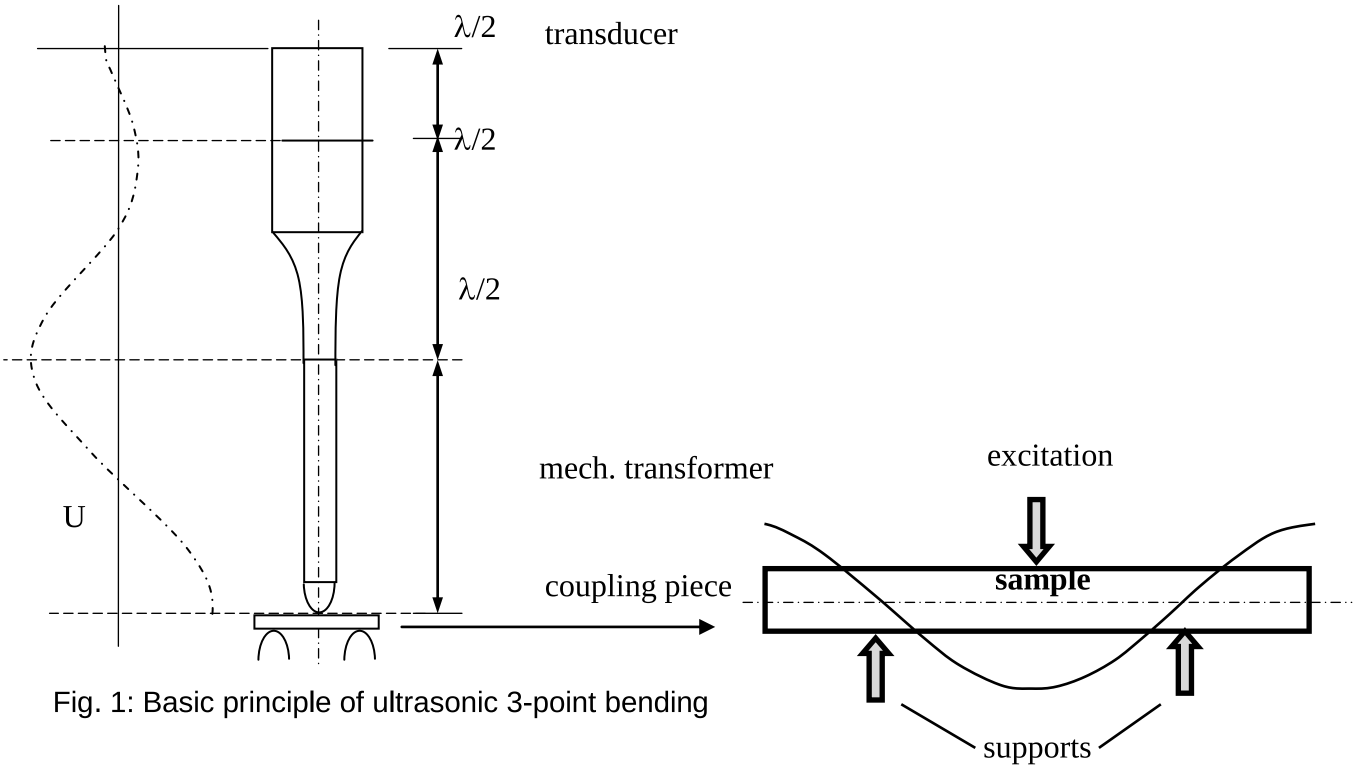
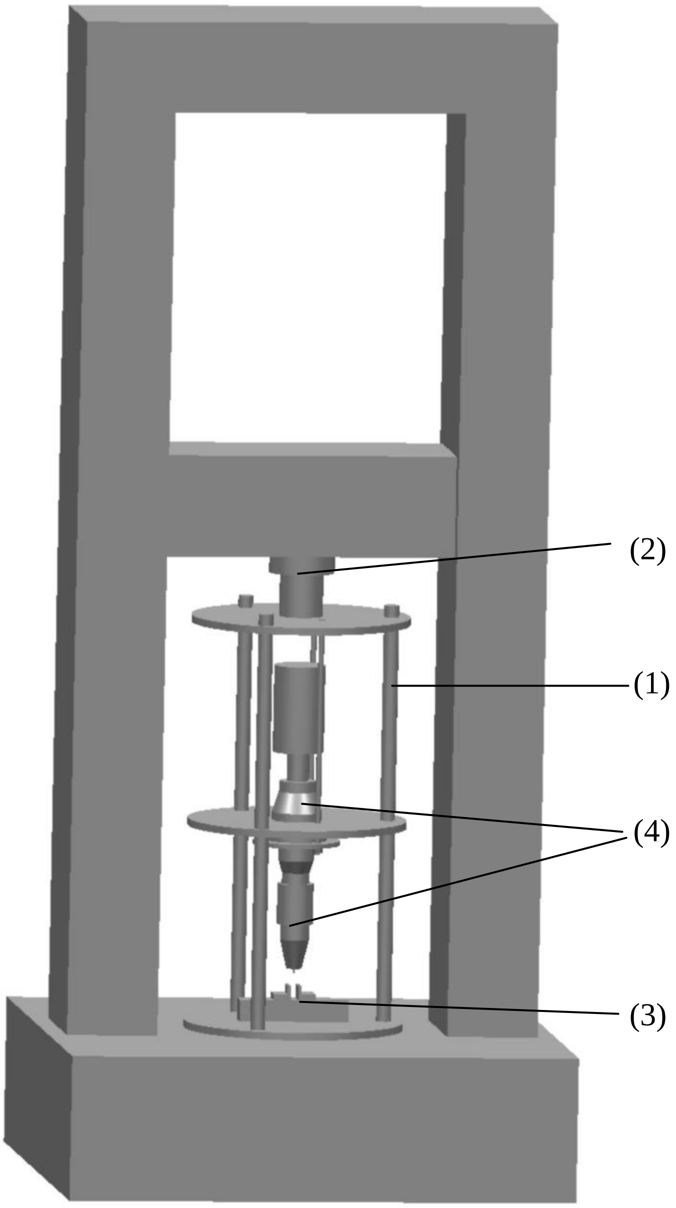
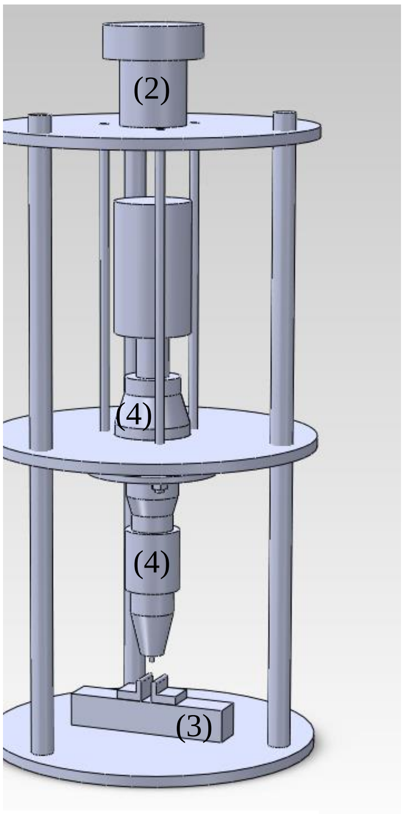


Fig. 1: Basic principle of ultrasonic 3-point bending

Design of a three-point bending set-up

The entire ultrasonic system is being inserted into a standard tensile testing machine by designing a special support structure as shown in fig 2. The support structure (1) is being attached to the crosshead resp. loading cell (2) of the testing machine. The three point bending device (3) is being adapted to support HM samples and is attached to the bottom of the machine. Further a special booster and a transformer (4) are designed which are attached to the supporting structure





Support structure (1))

Fig.2: Tensile testing machine with the bending fatigue set-up

WP 2a-Extension: ultrasonic three point bending system for fatigue studies of HM

Program: Construction of the machine (see above). Manufacture of the Samples. Surface preparation, check of Residual Stresses. Ultrasonic 3 PB testing of 1 Grade (E10)

Distribution of tasks:

- **WP 0**: Supply of 3PB Samples: **Industrial Partners** (E10: Ceratizit)
- Surface Preparation**: TUW/UW**
- Measurement of residual stresses of 2 samples: **CEIT**
- NPL will provide advice on the geometry and design of the three point bend specimens to help develop ways of ensuring a controlled and microstructurally representative fatigue fracture: **NPL**
- Fractography Key to development of the technique is to compare the fracture characteristics at high resolution of the new geometry to the cylindrical dumb bell geometry of previous project. NPL will examine fracture surfaces from a sample of the three point bend specimens both optically and in the scanning electron microscope. The fracture origins and the morphology of the fracture surfaces will be compared with similar observations of specimens of the same grade fractured in the UFTH2 project in order to contribute to a validation of the new test method. **NPL**
- Construction and adaptation of the testing equipment: Support structure, adaptation of the three-point bending stage, ultrasonic system. **TUW/UW** with subcontracting support from Company **Öfferl**
- Ultrasonic 3 PB testing of 1 Grade (E10): 1 S-N curve. **TUW/UW**

Cost of WP2a-3PB:
Contribution by industrial partners: cost not included here (See Item 8: WP 0)
Cost for TUW/UW – Öfferl (see Annex 2):

Construction and adaptation of the testing equipment:	10,080 € (incl. 20 % Austrian VAT)
Further costs are:	
Loading cell	1,680 € (incl.20% Austrian VAT)
Acoustic protection:	800 €
1,5 man month for testing + 20% overhead:	9,092€
Cost for NPL:	3,500€
Cost for CEIT:	1,000€
EPMA Mngt Fee:	2,600€
Total Cost of WP2a-3PB =	**28,752€**
Estimated duration of WP2a-3PB:	**ca. 7 Months in total**

Deliverables (TUW/UW, CEIT, NPL, UPC):
- Intermediate reports and presentations
- Final reports
- Measurement data points (xls file)

Total cost of WP 2a (Extension + 3PB) : **40,852€**

Gantt chart **WP 2a (Extension + 3 PB)**:

Estimation only (1 Unit= 0.5 Month)

1 Unit=0.5 Month :	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Work Package: "Extension"																				
Selection of materials (2 grades): E10 and SHM-C																				
Surface preparation		P	P	P	P			A	A	A	A									
Scanning Electron Microscope (SEM) studies						SEM	SEM					SEM	SEM							
Measurement of surface residual stresses						RS	RS					RS	RS							
Gigacycle fatigue testing -> 2 simpl. S-N curves																				
Damage evolution and interaction microstructure/crack…																				
Fractographic and Structural evaluation																				
Meeting/Reporting	M1													M2						M3
Work Package: "3PB"																				
Construction and adaptation of the testing equipment																				
Manufacture of test specimens: E10																				
Supply of material data																				
Surface preparation (G, P, A to be defined)						G?	P?	A?												
Measurement/supply of static properties, dynamic E									?											
Measurement of surface residual stresses																				
Gigacycle fatigue testing -> 1 S-N curve (E10)																				
Geometry Consulting																				
Fractographic evaluation																				
Meeting/Reporting	M1													M2						M3

Colour Codes:

TUW/UW	**Ind. Partners or Alternative**
CEIT	**Ind. Partners: SANDVIK or Alternative**
NPL	**Ind. Partners: CERATIZIT or Alternative**
UPC	
EPMA	

G: Grinding; **P**: Polishing; **A**: Annealing, **SEM**: Scanning Electron Microscope / Meetings for reporting (schedule to be confirmed): M1, M2, M3

Annex 2: Quotation of the company Öfferl

ÖFFERL
Messtechnik - Vertrieb & Engineering

Universität Wien
Physik Nanostrukturierter Materialien
Prof. Brigitte Weiss
Dr. Agnieszka Betzwar Kotas
Boltzmanngasse 5
1090 Wien

Kostenvoranschlag
Nummer: 012013
Datum: 10.01.2013

Sehr geehrte Frau Prof. Weiss, sehr geehrte Frau Dr. Betzwar Kotas,
anbei der Kostenvoranschlag für die Ultraschallbiegevorrichtung.
Bei dem Preis handelt es sich um einen geschätzten Preis, auf Basis unserer Besprechung und den von Ihnen vorgelegten Unterlagen.

Ultraschallbiegevorrichtung bestehend aus:

Aufhängevorrichtung mit äußerer Präzisionssäulenführung	(1)
Adaptierung einer 3-Punktbiegevorrichtung	(2)
Ultraschallsystem:	(3)
1x Sonotrode mit Druckspitze, 20kHz	
1x Halterungsbooster, 20kHz	
Obere Aufnahmeplatte inkl. Adapter für Kraftmessdose	(4)
Montage und Inbetriebnahme der Vorrichtung	

Ca. Preis excl. MWSt. 8.400,- €

Lieferzeit ca. 4Wochen nach Auftragserteilung und Materialverfügbarkeit

Mit freundlichen Grüßen
Leopold Öfferl

- 1 -

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