Revealing Properties of Structural Materials by Combining Regression-based Algorithms and Nano Indentation Measurements

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Abstract—The engineering expertise has been continuously increased within the decades such that very complex constructions are feasible, hence, high-performance structural materials are strictly required, which fulfill challenging performance profiles. Conventional material evaluation techniques have reached their performance limit such that new evolutionary approaches become increasingly important: A high-throughput screening approach has been proposed, which mainly operates on micro samples and applies multiple novel screening techniques to determine various characteristic values, which both leads to high volume of multidimensional data. This high volume allows to investigate many times more new candidates compared to conventional material development techniques. It is expected that the characteristic values reflect resulting material properties, which are not directly measurable due to chemical and physical limitations. Furthermore, the fact that no direct regularities between these characteristic values and resulting material properties are known, further increases the complexity of the data processing.

This work proposes a framework, which applies a state-of-the-art big data processing technique implementing a predictive function between characteristic values (determined on micro level) and material properties on macro level. In particular, a predictive function is implemented by orchestrating a kernel-based recursive least-squares algorithm, which processes micro hardness measurement (nano indentation) of micro samples to predict properties concerning the hardness as well as the yield strength, yielding to one elementary component of the high-throughput screening approach.

I. INTRODUCTION

The steady enhancement in engineering expertise has enabled constructions with a significantly increased complexity, which affects nearly all fields of everyday life. Powerful structural materials are required to realize such complex constructions to ensure a long durability even in detrimental applications, e.g., in automotive or in aerospace systems. This means that the involved structural materials have to fulfill certain performance profiles, i.e., the material has to exhibit a set of specific material properties like the hardness, the elongation or the yield strength. In fact, some of these material properties are known to correlate with each other, e.g., a linear relationship between hardness and strength as shown in [1], [2]. Standard screening techniques to determine the hardness and the strength differ in testing time and complexity. Thus,

finding relationships between material properties and swapping screening techniques can significantly reduce the testing effort. Furthermore, the specimen complexity, as required for tensile tests, can be avoided when using hardness measurements techniques, e.g. the instrumented nano indentation. Thus, using this kind of screening data to identify a suitable structural material with improved properties, forms an important objective.

Within the last decades, different alloys have been identified and have proven themselves for certain applications. In particular in combination with suitable heat treatments, those alloys obtain improved material properties. Adding further chemical additives to the conventional alloying constituents is one possible and commonly used technique to further improve the performance of the resulting material. For instance, the role of anion additives in the well-known nickel-cobalt system is investigated in [3]. These technical alloys allow to enhance specific properties, however, the high number of different alloying elements and, consequently, the reciprocal influences between these elements are not completely understood. Thus, this fact leads to unpredictable side effects, e.g., an increased tendency towards fatigue crack initiation and propagation [4].

A more promising strategy to improve the resulting properties aims to avoid any chemical additives but focuses on the primary alloy elements by varying the mass fraction (of an individual alloy element) as well as changing the manufacturing and treatment parameters, respectively. For instance, a naive approach can start from a known alloy system and adjust the phase transition while manufacturing and induce certain properties by postponed heat treatments. However, using such a conventional approach leads rather to slightly improvements than to significant breakthroughs. This is due to the low number of investigated influencing parameters, compared to the enormous imaginable search space, i.e., the huge number of possible different combinations of parameter sets. The identification of beneficial parameter sets is a time- as well as resource-intensive task.

To meet these challenges, a high-throughput screening technique is proposed in [5], which aims at exploring new structural materials fulfilling challenging performance profiles. In particular, a high-throughput flow, which operates at the micro level is established, which includes the manufacturing,

the post-processing and the screening of micro samples. These micro samples only enable the high-throughput character as well as the cost-effectiveness. More precisely, in [5] micro samples are manufactured and, subsequently, thermally and mechanically treated in a certain way, which is both specified by the process parameters. At the end, a set of process parameters is pursued, that can be used to fabricate a structural material with the desired performance profile.

By following the proposed scheme, a set of revolutionary screening techniques is applied on these micro samples, in which novel mechanical as well as electrochemical characteristic values, e.g., the conductivity or the X-ray diffraction, are measured. It is assumed that these characteristic values determined on micro level reflect certain macro-material properties, whose direct measurement is not possible on micro level and – so far– no correlation from material-scientific point of view is known. Thus, advanced data processing techniques must be applied to (1) project these screening data to the resulting material properties, (2) evaluate the current material properties against the desired performance profile and, especially, (3) in case of high deviation, identify a set of most promising process parameters, which leads to structural materials fulfilling the given performance profile. Invoking this proposed scheme leads to a significant amount of multidimensional data, which is due to the high-throughput character and the multiple measurements being applied within the screening process. In particular, processing this high volume of multidimensional data is one of the most crucial steps as outlined in [6].

This work proposes an approach to reveal unknown nexuses between screening data of micro samples and resulting material properties on macro level, which are not directly measurable. More precisely, experimental data from hardness measurements on micro as well as macro level [7] are exemplarily investigated to, finally, realize a predictive function that allows to project these characteristic values (experimental data) to hardness and elastic macro-material properties. This predictive function is one important step towards the evolutionary high-throughput approach [5] leading to superior structural material, as depicted in [6].

The structure of this work is as follows: Section II briefly introduces the high-throughput approach. Section III describes the basic principle of the investigated screening type, the instrumented nano indentation method and the invoked regression-based algorithm. An abstract overview of the proposed data processing frame is presented in Section IV. The experimental evaluation is presented in Section V. Finally, in Section VI some conclusions are drawn and an outlook on the further proceeding is given.

II. High-throughput Approach

This section describes the high-throughput screening approach [5], that aims to explore superior structural materials, which exhibit certain material properties such that a challenging performance profile is fulfilled, enabling new highly complex constructions.

In particular, the high-throughput approach identifies uninvestigated process parameter sets, i.e., chemical compositions in combination with specific parameters for the thermal and

mechanical treatment, which potentially lead to a structural material with enhanced material properties. These promising process parameters are determined by a data-driven algorithmic flow, which invokes a clever orchestration of different techniques within a feedback system.

This basic principle of this system is shown in Fig. 1 and consists of the following steps:

1) A set of process parameters forms the basis for the synthesis as well as the thermal and mechanical treatment of newly generated micro samples.
2) Different screening techniques are applied on the newly synthesized micro samples to determine characteristic values, which are fed into the predictive function to determine the expected material properties.
3) The predicted material properties are evaluated with respect to the desired performance profile by invoking certain relational operators.
4) If these properties deviate significantly from the performance profile, new process parameters are calculated by an optimization-based search engine. Otherwise, the process parameters are used to synthesize the superior structural material on macro level.
5) The output of the optimization-based search engine is coupled with a state-of-the-art experimental design procedure such that any prevalent inaccuracy as well as process variation (due to the fabrication) are addressed. This step determines a new promising set of process parameters, who controls the fabrication of further micro samples [cf. 1)], i.e., the feedback is approached.
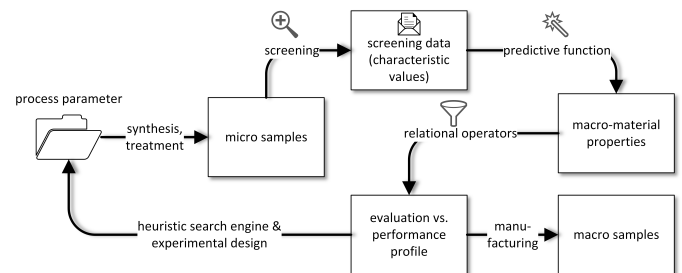

Fig. 1: High-throughput screening approach [5], [6]

Besides the stated Steps 1) to 5), the predictive capabilities are continuously evaluated and, if necessary, new correlated experimental data on macro level are conducted. These data provide new grid points in the function that increases the prediction quality. These new grid points are deliberately proposed by a postponed algorithmic flow [6], [8]. As presented in [6], it is planned that various screening techniques besides nano indentation are utilized to determine the characteristic values, for instance, impact tests, dilatometry, (cyclic) micro compression, micro magnetic and machining and X-ray diffraction. Furthermore, it is assumed that these characteristic values reflect important material properties concerning the hardness, the yield strength, the tensile strength, the fatigue strength, the elongation, the Young's modulus as well as the density of the resulting structural material.

One central and challenging aspect of this flow is to implement the predictive function, which has to cope with a

high volume of multidimensional data. Furthermore, a comprehensive analysis is required to identify nexuses between the characteristic values and predicted material properties. This is due to the fact that, so far, neither chemical nor physical relationships are known.

III. BASIC PRINCIPLES

This section presents some brief information about the utilized screening technique, namely the instrumented nano indentation. Furthermore, the basic principle of the regression-based algorithmic basis is described.

A. Nano Indentation Screening

The hardness of a material is defined as resistance against penetration. In comparison to standard hardness testing, micro hardness testing, e.g., the instrumented nano indentation, is applicable to very small or thin samples. The used testing device for the instrumented nano indentation, the Fischerscope® H100C [9], utilizes a Vickers pyramid made of diamond as an indenter [cf. Fig. 2(a)]. On top of that, the instrumented nano indentation offers the possibility to not only investigate the plastic but also the elastic material behavior while constantly measuring the penetration depth h at the given load F [cf. Fig. 2(b)]. The measured Martens hardness HM, the characteristic value of the nano indentation testing, is calculated by dividing the maximum load in the indentation curve by the contact surface, determined from the maximum penetration depth. The actual indenter's tip geometry (measured in SEM) is taken into account by using an additional shape correction.

Only small insecurities in the analysis of results exist in the instrumented indentation testing method, which is due to the fact that no optical measurement of the remaining indentation is required. With the available optical device, several samples can be located in advance and are automatically approached and tested with regard to a high-throughput experiment.

B. Regression-based Technique

The proposed framework focuses on experimental data, which have been generated by nano indentation experiments, i.e., by micro hardness measurements. On basis of these processed data, the following material properties are predicted:

- the hardness values HV following the Vickers method [10],
- the yield strength R_e, which is a characteristic value for the elastic-plastic behavior of a material determined out of tensile tests.

This framework provides a basis for further extensions, e.g., the support of further screening types or enhanced prediction capabilities of other material properties as intended in [6].

Definition 1. *Let $\mathcal{S}$ be the set of different screening techniques, which are applied on the micro samples, then the archetype of Ψ holds $\#\mathcal{S}$ dimensions spanning and let be $\mathcal{P}$ the set of investigated material properties, then the image holds $\#\mathcal{P}$ dimensions. Here, the different screening techniques can be interpreted as the available features, hence, the given dimensionality spans the feature space.*

Then, the predictive function Ψ is defined as $\Psi : \mathbb{R}^{\#\mathcal{S}} \to \mathbb{R}^{\#\mathcal{P}}$. Furthermore, a complete measurement of all screening techniques leads to a feature vector $v_s \in \mathbb{R}^{\#\mathcal{S}}$ and $v_p \in \mathbb{R}^{\#\mathcal{P}}$ describes the material profile, i.e., a set of exhibited material properties.

The core of this framework consists of the predictive function Ψ, which is formally stated in Definition 1: Ψ identifies nexuses between the screening data and the resulting material properties, which enables the actual prediction. Due to the fact that no exact relationships are known from a material-scientific point of view, an approximation of a given feature vector relating to the resulting material profile is required. In particular, Ψ implements a state-of-the-art regression-based technique, which invokes a derivative of the frequently used Least-Squares algorithm, more precisely, a *Recursive Least-Square* (RLS) algorithm. This class of algorithms are known to work well in case of continuous data generation, for instance, in the field of communication or signal processing [11], which are both comparable to the data generation of the intended high-throughput approach [5].

The huge resulting feature space with $\#\mathcal{S} > 40$ is a further aspect, which has to be considered. To meet this challenging computation, kernel-based approaches have been introduced by Vapnik in [12], who has shown that the computational problem is easier when the number of dimensions is increased even more. However, these initially proposed approaches are not suitable for continuously data processing. Such a continuity is given by the high-throughput approach, for instance, by the continuously evaluation of Ψ and the increasing number of grid points. This would lead to a complete re-computation of the kernel-function in a conventional approach [12], which is not feasible in reasonable time due to the high data volume.

For coping with this challenge of continuous data streams, the authors in [13] have proposed an online *Kernel-based Recursive Least-Squares* (KRLS) algorithm, which extends the conventional RLS algorithm by utilizing a *Mercer's Kernel* [14]. For instance, prominent candidates are polynomial as well as *Gaussian* kernel-functions. Thus, such a KRLS algorithm fulfills completely the requirements for implementing the regression.

IV. IMPLEMENTATION

This section presents some technical implementation details of the developed data processing framework. As already stated in Paragraph III-B, the framework bases on a KRLS algorithm, i.e., a kernel-function is built by processing experimental data corresponding hardness measurements on micro and macro level.

In [6] an outlook is given on the intended dimensionality of the screening data as well as the material properties, although it is necessary that the algorithmic base covers the following scenarios:

- At least some spare material-scientific nexuses are known and have to be considered, e.g., a relationship between specific characteristic values and certain macro-material properties.
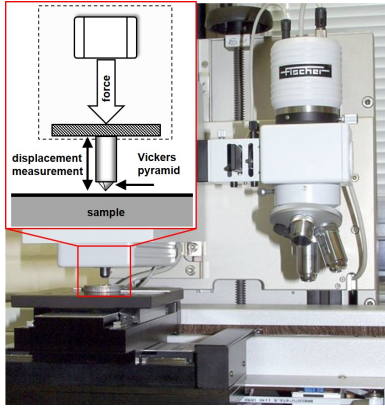
(a) Testing device Fischerscope® H100C
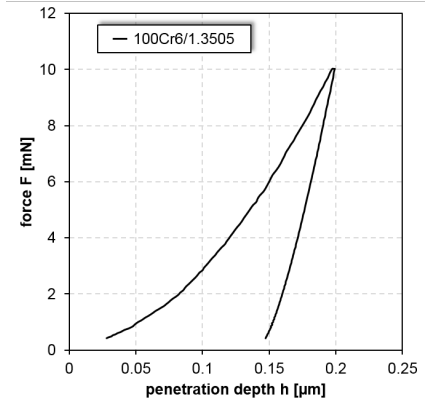
(b) Force-penetration depth diagram in the micro section of a standardized bearing ball, 1mm (bearing steel SAE52100 (German grade 100Cr6))

Fig. 2: Instrumented nano indentation

- Multiple screening techniques provide potentially contradictory information related to a single macro-material property.
- An external data analysis leads to hypotheses, which have to be considered or evaluated, respectively.
- The level of confidence is not equally distributed between different screening techniques.

To tackle these requirements, it is necessary that screening data from different screening techniques can be evaluated individually. This means that the influence, relating to macro-material properties, of a subset of features being $\mathcal{S}_i \subset \mathcal{S}$ has to be adjustable. Consequently, the proposed framework utilizes not just a single kernel-function $\mathcal{K}$ but allows to set up n kernel-functions $\mathcal{K}_1, \ldots, \mathcal{K}_n$. Each $\mathcal{K}_i$ is defined by a pair $(\mathcal{S}_i, \mathcal{P}_i)$ with $\mathcal{S}_i$ a subset of features (as stated above) and $\mathcal{P}_i \subset \mathcal{P}$ of macro-material properties.

Such an exemplary kernel-function mapping is visualized in Fig. 3: The upper box represents the possible screening techniques (feature space), including the single instrumented screening techniques (features), for instance, the micro machining, which provides different characteristic values (sub-features). The box at the bottom represents the macro-material properties, e.g., the hardness or the yield strength, which should be predicted. The central box represents the kernel-function database, which is the important algorithmic part for the actual prediction. This database stores two exemplary functions: The left one processes two characteristic values – one measured by the nano indentation test and one by the impact test – and predicts macro-material properties concerning the yield strength and the hardness. Analogously, the right one processes one characteristic value from the impact test as well and one from the X-ray diffraction measurement and predicts properties relating to the hardness, the tensile strength as well as to the Young's modulus.

All kernel-functions are aimed to be stored in a serialized way, e.g., within a database. In fact, this separation can also take place on basis of single characteristic values of a single screening technique if necessary. Besides, the framework also provides to set up two kernel-functions $\mathcal{K}_1$, $\mathcal{K}_2$, which are both defined by the same $\mathcal{S}_i$ as well as $\mathcal{P}_i$. For instance, this is required in the case that screening data for micro
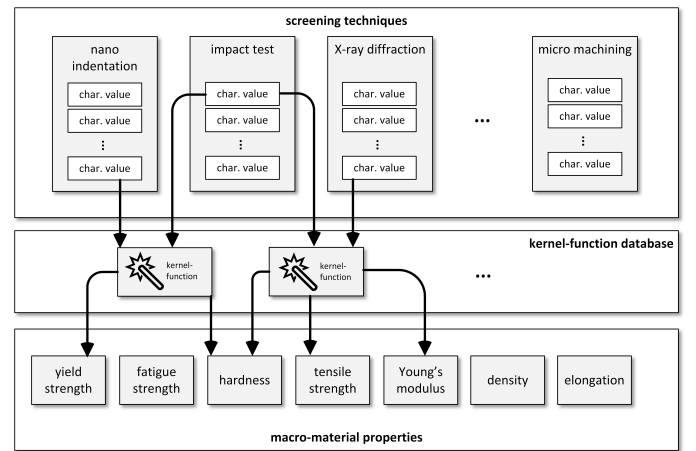

Fig. 3: Kernel-function mapping

sample, which have been manufactured by completely different alloying system, should be evaluated separately.

The complete data flow of the proposed framework, which is shown in Fig. 4, is as follows:

1) A user starts the main program to predict resulting macro-material properties.
2) If required, for instance, the kernel-function database is still empty, the data parser is invoked to parse the macro data once.
3) After the control unit has been configured as determined by the user-defined configuration,
4) these macro data are used to built up the kernel-functions, which have been already prepared by the control unit.
5) The data parser processes the screening data and, subsequently, these data are used for prediction.
6) The prediction engine loads the specific kernel-functions from the kernel-function database and invokes them on the given data.
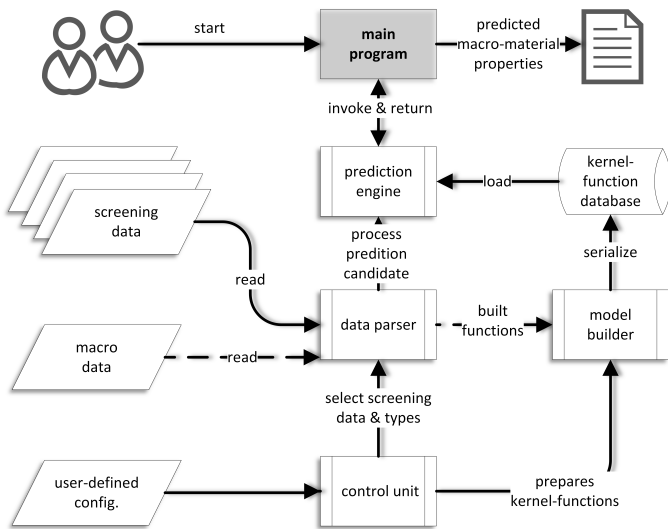7) Finally, the predicted macro-material properties are returned.

Fig. 4: Prediction Framework

V. Experimental Results

This section describes the experimental evaluation of the proposed data processing framework, which identifies nexuses between screening data and resulting material properties. In particular, this framework allows to predict resulting hardness, strength as well as elastic properties of structural materials by analyzing nano indentation screening data.

All experiments were executed on an *Intel Xeon E3-1270v3 3.5 GHz* processor with *32 GB* system memory. The proposed framework is written in C++ and includes the dlib [15] library. The nano indentation (cf. Paragraph III-A) data were measured by a Fischerscope® H100C [9] device.[1] Different sets of process parameters, which define a chemical composition and a specific heat treatment, were used to fabricate the micro samples.

Three different classes of micro samples were considered:

1) Embedded, standardized bearing balls [16], [17],
2) Embedded, spherical micro samples with a diameter from $300\mu m$ up to $1000\mu m$,
3) Micro samples fabricated by an additive, laser-based technique [18].

Samples 1) and 2) are both manufactured by using a 100Cr6/1.3505 alloying system. The heat treatment for 1) was executed by standardized parameters, i.e., according to the literature, and for 2) slightly varying heat treatment parameters were used. For generating samples 3), a C15/1.0401 base material is used and mixed by selective laser melting with a powdered iron-based master alloy (components according to Table I) in order to reach a material composition like 100Cr6. However, the generated composition is different from the target material 100Cr6 due to the powder components, e.g., the micro samples' nickel content is still higher than intended. Additionally, varying process parameters during the selective laser melting method, such as laser power, lead to different heat treatments for samples 3).

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[1] Hereby, the measured HM value is considered as the characteristic value.

TABLE I: Chemical alloy components [19]

C %	Mo %	Ni %	Si %	Mn %	Cr %	P %	S %	Fe %
0.012	2.27	13.1	0.56	0.31	16.5	0.017	0.007	Balance

Due to incomplete screening data of macro level measurements, which have not been conducted so far, reference values from the literature [20], [21] are used to complete the data set for samples 1) and 2). Furthermore, the validity of these literature values is also assumed for samples 3), although, the chemical composition still differs due to instability within the laser melting process. Within the evaluation all three classes of micro samples have been investigated, i.e., the screening data of the instrumented nano indentation (cf. Paragraph III-A) were fed into the prediction framework. More precisely, the measured Martens hardness HM as well as the maximal penetration depth h.

Table II presents the final evaluation results while measuring the following data:

HV Hardness following Vickers in HV,
R_e yield strength in MPa and
t overall run-time in sec.

In general, two different kinds of values are measured: At first, statistical value for actual prediction (the predicted macro-material property) and, secondly, statistical error data about the prediction itself. The error data have been measured by validating the predicted value with respect to the literature data concerning the material properties of the 100Cr6 alloying system. More precisely, the following values are calculated:

$mean$ mean value of the predicted property,
med median value of predicted property,
dev deviation of the predicted property,
me mean error value of the prediction,
de default error value of the prediction and
rms root mean square error value of the prediction.

As clearly visible, the prediction for samples 1) works best. This is due to the fact that these samples were manufactured and heat treated accordingly to the reference values from the literature [20], [21]. Thus, both the HV as well as R_e value converges to the expected literature values for 100Cr6 with $HV = 800HV$ and $R_e = 2030MPa$, respectively. In addition to this, the mean error value for both predicted properties is negligible with 3.0 and 8.0.

In comparison to that, the prediction for samples 2) achieves a well approximated hardness prediction (in sense of the error values). However, while predicting elastic properties, the error values strongly increase. This potentially indicates that the significant different heat treatment between 1) and 2) has changed the elastic character much stronger compared to the hardness.

As expected, the prediction for samples from 3) holds a higher mean error value of 17.0 (HV) and 60.0 (R_e) and, furthermore, even a higher default error compared to 1). This is

TABLE II: Evaluation: Prediction of macro-material properties

Class of sample	HV [HV]						R_e [MPa]						t [s]
	mean	med	dev	me	de	rms	mean	med	dev	me	de	rms	
1)	799.0	799.2	44.7	3.0	485.5	58.7	2041.0	2126.5	366.9	8.0	247.7	58.7	< 1
2)	799.0	799.2	0.16	61.0	1123.8	108.5	1647.0	2065.3	772.4	52.0	1220.4	107.7	< 1
3)	692.0	699.4	176.9	17.0	531.7	58.5	1567.0	2100.5	850.1	60.0	1531.3	112.43	< 1

due to the fact, that the reference values are taken from the literature and do not fit completely to the additive manufactured samples 3) as already mentioned above. Additionally, only one single grid point per material property could be used for building the predictive-function. Consequently, the prediction quality has to be further improved, particularly while varying the heat treatment. This can be achieved by conducting more experimental data, which consist of correlated micro and macro hardness experiments.

In general, for both prediction types in all conducted experiments, it can be noted that the default error value is quite high, which indicates that the set of processed data must be further enhanced [22]. In fact, due to this exemplary character, the amount of processed data was limited, although, the root mean square error value indicates already that this prediction framework works.

VI. Conclusions & Future Work

This paper proposed a data processing framework, which is capable to predict resulting material properties concerning the hardness as well as the yield strength of structural materials. In particular, experimental data are processed, that have been conducted by micro and macro hardness measurements. The conducted experiments prove that the concept of this prediction framework works and, by processing more data, it is expected that the prediction quality can be further improved. The proposed framework strongly contributes to the proposed high-throughput approach for structural material development [5] and provides an algorithmic structure, which can be easily extended for further screening techniques and material properties, as required in this context [6].

Future work aims to increase the number of input as well as output dimensions of the predictive function, i.e., an increased number of screening techniques will be supported and, furthermore, additional material properties will be also taken into consideration. Additionally, it has to be stated that feature vectors are potentially incomplete, for instance, it is not possible to apply two different destructive screening types on a single sample. Thus, the predictive function has to process incomplete feature vectors as well without causing any adverse impact on the kernel-function.

VII. Acknowledgment

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