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高通量统计映射表征技术研究进展及其应用

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摘要

实际材料是非均匀的、多元的、复杂的，任何材料在特定尺度下都不可能是绝对均匀的或者绝对规则的，可视为“自然芯片”，一个材料每个位置上的成分、组织结构和性能都存在着微小差异，而这种微小差异的组合形成了材料整体性能，本文综述了基于材料非均匀性本质的高通量统计映射表征技术的研究及应用进展。高通量统计映射表征技术采用一系列跨尺度快速表征技术，从宏观到微观逐级分析，快速获取实际大尺寸试样每个位置上的成分、组织结构和各种性能参量的数据集；通过准确的位置坐标信息，将这些数据集按照点对点的对应关系，形成组合映射点阵数据仓库；根据材料研发的设计需求，从数据仓库中筛选出符合要求的目标区间内的映射数据集；通过对目标区间内映射数据集的统计解析，筛查出接近设计需求的映射数据集，再经过反复验证最终获得反映材料性能的基本单元（组）；通过确定的工艺优化指向参量，验证这些基本单元（组）在介观尺度的组装，最终建立微观-介观-宏观-常规试样-构件跨尺度的成分-组织结构-特性间的量化相关性。高通量统计映射表征技术已在各种钢铁、高温合金、镀层板、铁硅合金等材料体系得到应用，有效地指导了工艺的优化及材料的改性。

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1. 引言

1995年，项晓东等[1]在*Science*封面文章上首次发表“组合材料芯片”的研究，在1块基片上实现128种不同组分的新材料的同时集成生长和表征，实现高通量的材料筛选及“材料相图”的系统描绘。此后，类似的适用于不同领域的高通量材料合成及表征技术也相继得到发展并被成功应用，如采用“扩散多元节”方法实现了结构材料的加速设计[2]，采用高通量薄膜生长技术实现分立及连续组分半导体材料的筛选[3]，采用喷墨递送系统实现粉体组合材料的筛选[4]，更有多种高通量实验技术实现了材料的成分[5]、组织结构[6]、电化学性能[7]、催化性能[8]、电磁性能[9]、磁性能[10]、光学性能[11]、热性能[12]、力学性能[13]等方面的快速表征，使高通量筛选未知新材料的周期大大缩短。采用传统方法需要花费数年时间才能完成的工作，通过高通量实验技术在极短的时间（如一星期）内即可完成，实现了革命性的突破。然而这种“组合材料芯片”技术也有不足之处，就是芯片的制备工艺往往与材料的实际生产工艺相差甚远，还需要进行诸多的生产工艺研究才能将实验室里研发的材料转化为真正意义的材料。王海舟[14,15]首次提出高通量统计映射表征新方法，该方法是

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引用本文: Haizhou Wang, Lei Zhao, Yunhai Jia, Dongling Li, Lixia Yang, Yuhua Lu, Guang Feng, Weihao Wan. State-of-the-Art Review of High-Throughput Statistical Spatial-Mapping Characterization Technology and Its Applications. *Engineering*, https://doi.org/10.1016/j.eng.2020.05.005

在原位统计分布分析（original position statistical-distribution analysis, OPA）表征技术的基础上发展而来，基于材料非均匀性本质的认知，对实际样品进行高通量、跨尺度的统计分布表征，快速获取具有准确位置对应关系的从宏观、介观至微观的成分、组织结构和各类性能的海量数据集合，并建立各数据集合的映射关系，获取成分、组织结构和性能的相关性，快速筛选出性能优异的基本单元组，从而指导现有材料的改性和实际生产工艺优化；另外，通过对海量映射数据集的机器学习，可将这些筛查出的材料基本单元组，从微观至宏观逐级反演重构，实现整体宏观材料组成、结构和性能的优化组合，从而完成新材料的逆向设计，加快实现新材料的发现。因此，具有非均匀性本质的实际材料也被视为具有多种组合的“自然芯片”，由于这种“自然芯片”来源于实际生产工艺，其对现有材料的改性和生产工艺的优化具有重要的指导意义。

2. 高通量统计映射表征技术

2.1. 材料的非均匀性本质

实际材料是复杂而又多元的，其本质具有非均匀性，任何材料在特定尺度下都不可能是绝对均匀的。因此，材料不同位置上的成分、组织结构和性能都存在着微小差异，而这种非均匀的成分和组织结构通过排列和组合，构成了材料的整体性能。与人类基因类似，材料也存在着某种能够直接反映材料特性的最小物质单元——基本单元。针对不同材料，其基本单元也是非唯一的，它可以是组成物质的任何自然存在的原子、分子、离子的粒子，也可以是这些粒子组合而形成的相、团簇、基团、单元、晶粒等，将相同的或不同的基本单元以某种工艺或技术结合起来也就形成了具备某种特性的基本单元组，甚至最终材料。而这种针对基本单元（组）的结合工艺或技术可称为“组装”，由于不同材料及其工艺的复杂性和多样性，其基本单元的“组装”方式也具有多样性的特点。材料基因工程研究从某种意义上讲，就是高通量的制备、表征、筛查、组装和反复验证这种按需设计的目标基本单元（组），从而建立微观-介观-宏观-实际构件跨尺度的组成-结构-性能的相关性，指导新材料的研发和材料的改性，实现研发周期和研发成本双减半。王海舟[16]等采用原位统计分布分析表征技术研究高温合金压气机盘锻件中的铌分布时发现，盘锻件的铌元素在下模冷模影响区和锻件中心两个部位分布不均匀，导致整体盘锻件质量不合格，但在分析结果指导下，对盘锻件加工时去除了不均匀部位，使得加工成品的质量达到合格标准。非晶合金在不同方向上的物理性质是相同的，这是由于其原子结构在较大尺度上看是无序且各向同性的，但已有诸多研究表明非晶合金在纳米甚至微米尺度上的结构和动力学是非均匀的[17–24]。因此，材料的非均匀性客观存在，但如果能够基于这种本质，更加深入、清晰地认知材料的基本单元（组），那么就能够更快、更好地设计和开发出性能更加优异的新材料。

2.2. 高通量统计映射表征技术方法

高通量统计映射表征方法是基于材料非均匀性本质，通过材料的跨尺度表征，获取材料中数以万计微点阵上不同组成、结构和性能参量，依据材料各原始位置建立各参量集合之间的统计映射模型，再根据统计映射模型筛查材料特性基本单元（组），结合高通量计算形成材料数据库，进行新材料的优化设计，从而指导材料的改性、工艺优化和新材料的发现，如图1所示。

高通量统计映射表征的流程如图2所示，采用一系列跨尺度快速表征技术对实际材料从宏观到微观逐级分析，快速获取非均匀的大尺寸试样上不同位置上的成分、组织结构和各种性能参量的数据集；按照确定的位置坐标信息将这些数据集进行组合映射，并形成数据仓库；根据材料研发的设计需求，从数据仓库中筛选出目标区间内的映射数据集；通过对目标区间内映射数据集的统计解析，如各参量范围频度统计解析、各参量相关性统计解析、异常值统计剔除、规律符合性科学判定等，筛查出接近设计需求的映射数据集；再经过反复验证最终获得反映材料性能的目标基本单元（组）；通过确定的工艺优化指向参量，验证这些目标基本单元（组）在介观尺度的组装，最终建立微观-介观-宏观-常规试样-构件跨尺度的成分-组织结构-特性间的量化相关性。

高通量统计映射表征方法就如同走迷宫时采取多路径并行试探法，可以大大提高新材料发现的效率，颠覆现有材料“单一试错法”的研发模式，为新材料快速研发开辟新的商业模式，构建新材料低成本、短周期研发的“高通量试错法”材料研发创新体系。

2.3. 跨尺度原位统计分布分析技术研究

原位统计分布分析是指较大尺度范围内（cm^2）各化学组成及其形态的定量统计分布分析技术。它包含化

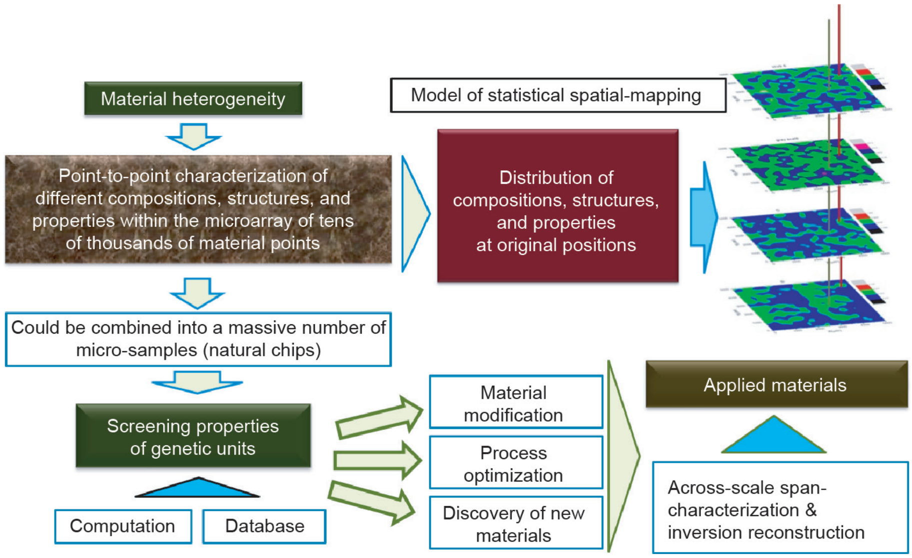


图1. 基于实际材料非均匀性的高通量统计映射表征技术。

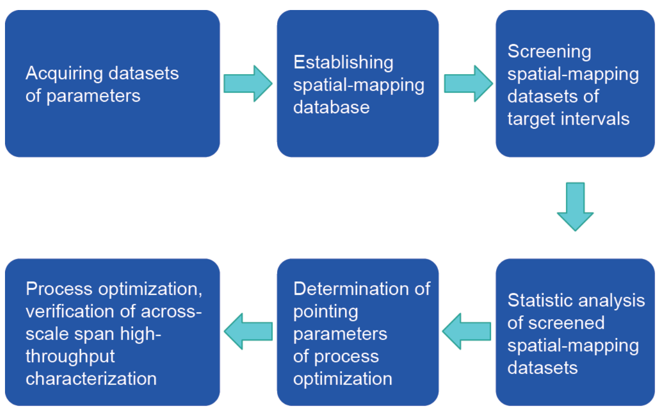


图2. 高通量统计映射表征的流程。

学组成的位置分布信息、含量分布统计信息以及状态分布统计信息，按区域划分又可分为一维原位统计分布分析、二维原位统计分布分析以及三维原位统计分布分析等[25–32]。目前，原位统计分布分析技术已应用于成分、组织结构和力学性能等方面的表征，发展出如火花源原位统计分布分析技术（简称火花原位技术）、激光诱导击穿光谱原位统计分布分析技术（简称激光原位技术）、激光剥蚀电感耦合等离子体质谱原位统计分布分析技术（简称激光剥蚀原位技术）、微束X射线荧光光谱原位统计分布分析技术（简称微束荧光原位技术）、全视场金相组织原位统计分布分析技术（简称全视场金相原位技术）、大尺寸高通量扫描电镜原位统计分布表征技术（简称电镜原位技术）、流体微探针应力应变原位统计分布表征技术（简称流体微探针原位技术），并且研发出一系列具有自主知识产权的仪器装置。

2.3.1. 火花源原位统计分布分析技术及装置

作为材料宏观统计分布表征的主要手段，火花源原位统计分布分析技术以单次火花放电理论及信号分辨提取技术为基础，开发出火花微束（探针）技术、无预燃连续激发同步扫描定位技术，并据此获得数以百万计与材料原始位置相对应的各元素原始含量及状态信息，用统计解析的方法定量表征材料的偏析度、疏松度、夹杂物分布等指标。火花源原位统计分布分析技术一方面能够获取金属材料较大尺度范围内（几百平方厘米）各成分的位置分布、状态分布及定量分布的准确信息[33]；另一方面由于每根非约束“探针”的激发束斑尺寸为1~10 μm，因此信号能反映出非常精细的元素状态信息。可以认为，火花源原位统计分布分析技术是一种能在宏观尺度下反映微观状态的跨尺度表征技术。首台金属原位分析仪OPA-100于2002年成功研制，申请了6项国内外发明专利[34–40]，并获2008年国家技术发明奖二等奖[41]，目前已升级至第四代产品（图3）。

火花源原位统计分布分析技术对于大尺寸金属材料成分偏析和夹杂物定量表征的应用已日臻成熟，在指导生产工艺优化方面发挥了重要作用。在成分偏析表征方面，已成功应用于各种连铸坯、帘线钢、船板钢等碳钢、不锈钢材料表征[42–81]。例如，李冬玲等[82]对35号碳素钢圆坯的研究表明，电磁搅拌工艺生产的铸坯边部产生白亮带，其C、Si、Mn、P元素存在明显的负偏析，是造成晶粒组织和维氏硬度（Vickers hardness, HV）分布不均匀的主要原因（图4）。王文龙等[83–85]还对铝合金和黄铜类的有色金属中的成分偏析及质量缺陷进行了研究。

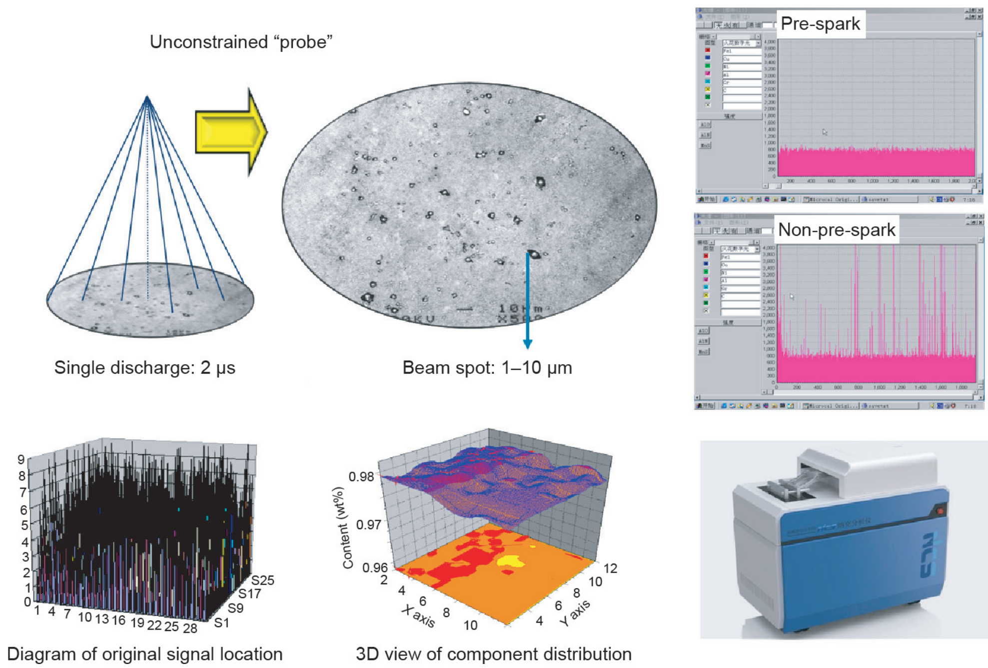


图3. 火花源原位统计分布分析技术原理及装置。

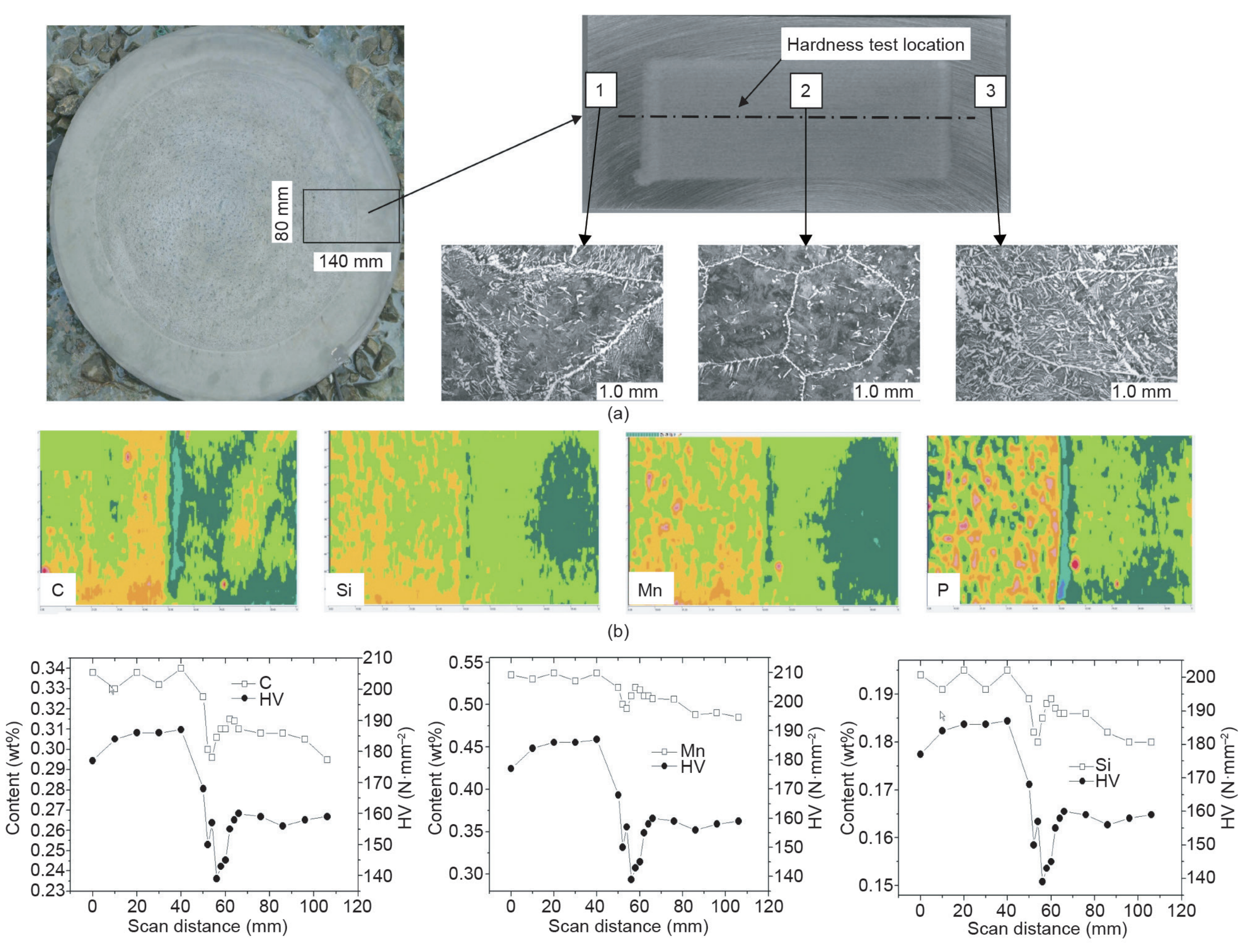


图4. 火花源原位统计分布分析技术在电磁搅拌圆坯成分偏析表征中的应用。（a）样品的分析区域106 mm×52 mm；（b）分析区域元素含量二维分布图；（c）C、Mn、Si的成分分布与维氏硬度的相关性。

在夹杂物定量表征方面，杨志军等[86–89]通过对单次放电异常火花数量的统计解析，建立了钢中锰系、铝系、钛系、硅系等夹杂物的含量及粒度分布分析方法；火花源原位统计分布分析技术已成功应用于多种碳钢、不锈钢、重轨钢、大梁钢、齿轮钢、高压锅炉管钢等金属材料中的夹杂物统计分布分析[90–107]。例如，罗倩华等[108]采用火花源原位统计分布分析技术对不锈钢连铸板坯横截面全尺寸表征（样品A~N），发现低倍试验中样品出现白亮带的边部位置铝钙夹杂比例略高，且小颗粒夹杂比例略多，而中部区域铝氧夹杂比例略高，且大颗粒夹杂比例略多，与扫描电镜分析结果一致，最终获得从边缘至中心的整个板坯的Al系夹杂物分布图（图5）。

2.3.2. 激光诱导击穿光谱原位统计分布分析技术及装置

激光诱导击穿光谱原位统计分布分析技术基于高能激光束作用在材料表面上产生的原子发射光谱信号进行定量分析，束斑尺寸为微米至毫米，其具有非接触分析、微区分析、深度分析等优点，能够进行任意点扫描、一维线扫描、一维深度分析和二维面分析，能够实现在样品表面很好的定位，是材料介观至宏观跨尺度表征的有效手段[109–111]。首台商品化激光原位分析仪LIBS-OPA100于2010年成功研制，申请多项发明专利[112–115]，目前已升级至第二代产品（图6）。

激光诱导击穿光谱原位统计分布分析技术在小尺寸样品的成分偏析表征方面具有优势，张勇等[116–122]采用LIBS-OPA100对中低合金钢板坯、帘线钢盘条、X80管线钢堆焊融合区、电迁移金属钆棒等小样品中多种组分的分布情况进行了分析，通过成分偏析表征指明了生产工艺存在的问题。图7为X80管线钢堆焊区激光原位成分分布表征与显微组织和维氏硬度分布的相关性研究。

激光原位技术在样品缺陷分析方面进行了许多研究，赵雷等[123–126]采用LIBS-OPA100对汽车板、冷轧热镀锌板表面的各种形状缺陷进行了线扫描、面扫描和深度分析。研究结果表明，缺陷部位伴有元素的偏析，绝大多数是由于生产过程中带入了保护渣所致，对镀层板生产工艺的改进具有指导意义。图8为某汽车板缺陷的深度及线扫描研究。

近年，激光原位技术在夹杂物分析方面也取得了一些成果，杨春等[127–130]通过研究发现激光光谱的异常信号个数反映夹杂物个数，而异常信号强度的高低则反映夹杂物尺寸大小，并成功分析了钢中的酸不溶铝、MnS夹杂和Si-Al-Ca-Mg复合夹杂的含量，分析结果与传统湿法分析结果相吻合。图9是激光原位技术在MnS夹杂物定量方面的研究。

2.3.3. 激光剥蚀电感耦合等离子体质谱原位统计分布分析技术及装置

激光剥蚀原位技术的原理是微束聚焦的激光可将样品逐层剥蚀并气化，以气溶胶的形式通过惰性气体输送至电感耦合等离子体（inductively coupled plasma, ICP）源离子或原子化，再经质谱仪进行定量分析。该技术的激光束斑为微米级、检测限低、灵敏度高，适于异形或小样品低含量及痕量组分的面分布统计分析，是材料介观至宏观跨尺度表征的另一有效手段。2008年，激光剥蚀原位技术成功开发，并应用于多种球扁钢、镀锌钢管、焊管、高温合金涡轮叶片、金属镝棒、管线钢裂缝、冲击断口等小尺寸样品异形表面的统计分布表征，反映各成分的位置分布、统计偏析度、最大偏析度及其与材料质量间的相关性[131–147]。2015年全套激光剥蚀原位表征设备（包括激光烧蚀进样装置和电感耦合等离子体质谱仪）实现自主知识产权的商品化，见图10，申请发明专利两项[148,149]，并获得2015年中国分析测试协会设立的北京分析测试学术报告会暨展览会（Beijing Conference and Exhibition on Instrumental Analysis, BCEIA）金奖。图11是激光剥蚀原位技术在定向凝固高温合金涡轮叶片的统计分布表征研究，结果表明，低熔点元素在多晶带区域析出造成叶片缺陷[150]。

2.3.4. 微束 X 射线荧光光谱原位统计分布分析技术

微束荧光原位技术是采用毛细管透镜将X射线聚焦为直径约20 μm的小束斑，基于微束X射线荧光光谱对材料表面的化学成分进行无损面扫描检测，并对获取的大量数据进行统计分布分析。微束荧光原位技术是一种无损的检测方法，既提高了分辨率，又没有过多地损失荧光强度，扫描范围可达厘米级，是材料介观至宏观跨尺度表征的高效表征手段。具有自主知识产权的原型机于2017年成功研制，见图12。杨丽霞等[151]采用微束荧光原位技术对耐候钢薄板坯的微区成分偏析情况进行了统计分布表征，结果表明裂缝区域Ti、Mn、P、S的偏析可能是引起材料开裂的主要原因，见图13。李冬玲等[152]采用微束荧光原位技术对不同工艺的高温合金中的成分进行了统计分布表征，结果表明，热处理工艺

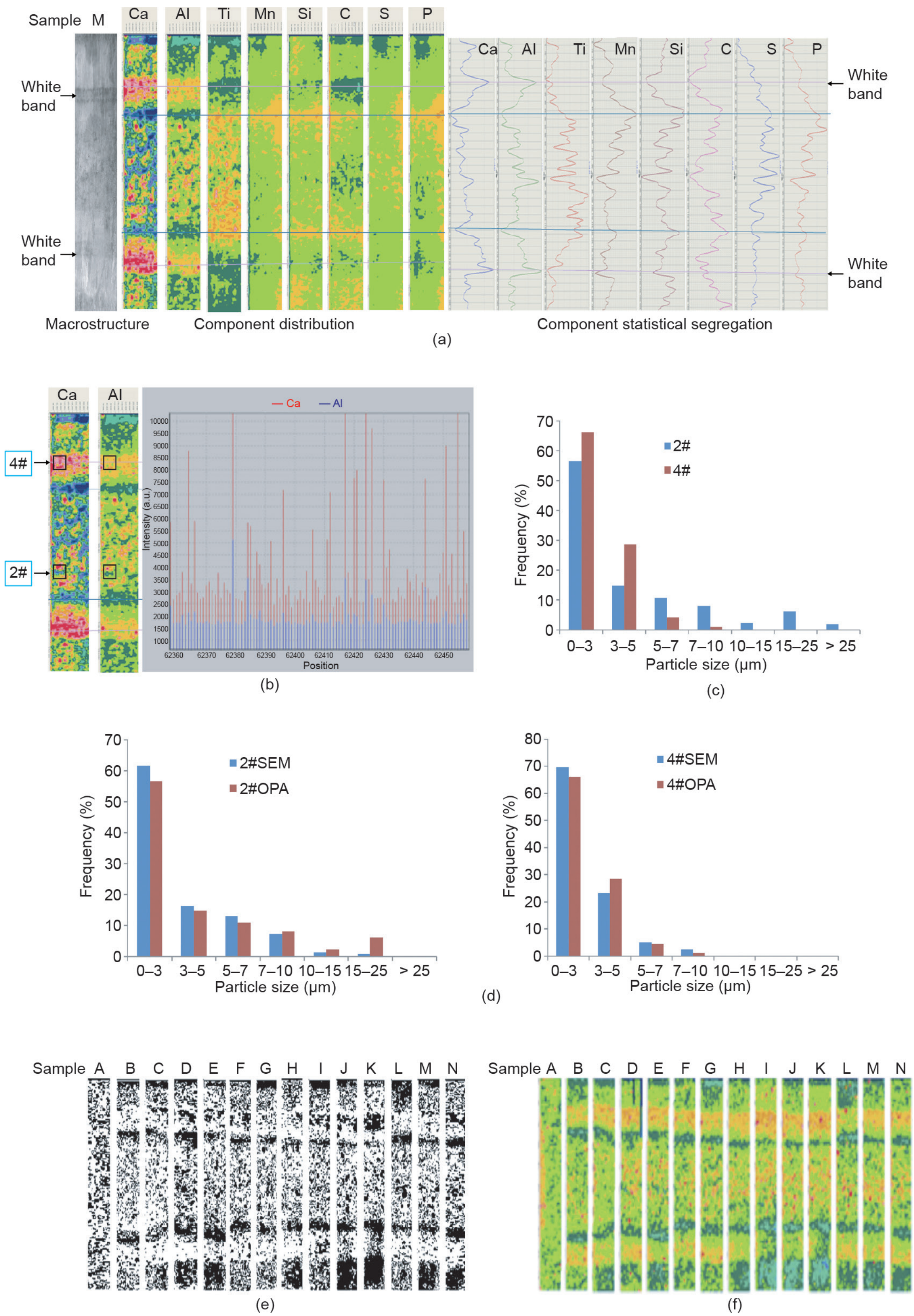


图5. 火花源原位统计分布分析技术在连铸板坯夹杂物表征中的应用。(a) M号样品的低倍、成分分布及统计偏析度分析；(b) Al、Ca元素异常火花通道合成图；(c) 2#和4#区域铝系夹杂物粒度分布图；(d) 2#和4#区域采用SEM和OPA两种方法获得的铝系夹杂物粒度分布图；(e) 整个板坯Al系夹杂物分布图；(f) 整个板坯Al元素成分分布图。

有效改善了Nb、Ti、Mo、W元素的分布均匀性，最大偏析度显著减小，见图14。

2.3.5. 全视场金相组织原位统计分布分析技术

全视场金相原位技术基于全自动扫描型金相显微镜快速获取样品全表面范围的金相组织图谱，以及位置信息，并拼接成一个具有精确位置信息的完整图像，通过对图像每个位置上原始数字信号（灰度值）的统计解析，实现各类组织结构（如疏松、裂纹、缩孔、缺陷、晶粒、析出相、夹杂物等）的自动鉴别和定量统计分布表征。全视场金相原位技术通过对试样全范围内的组织结构定位统计分析，解决了人为选择视场时具有主观性、随机

图6. 激光原位分析仪LIBS-OPA100。

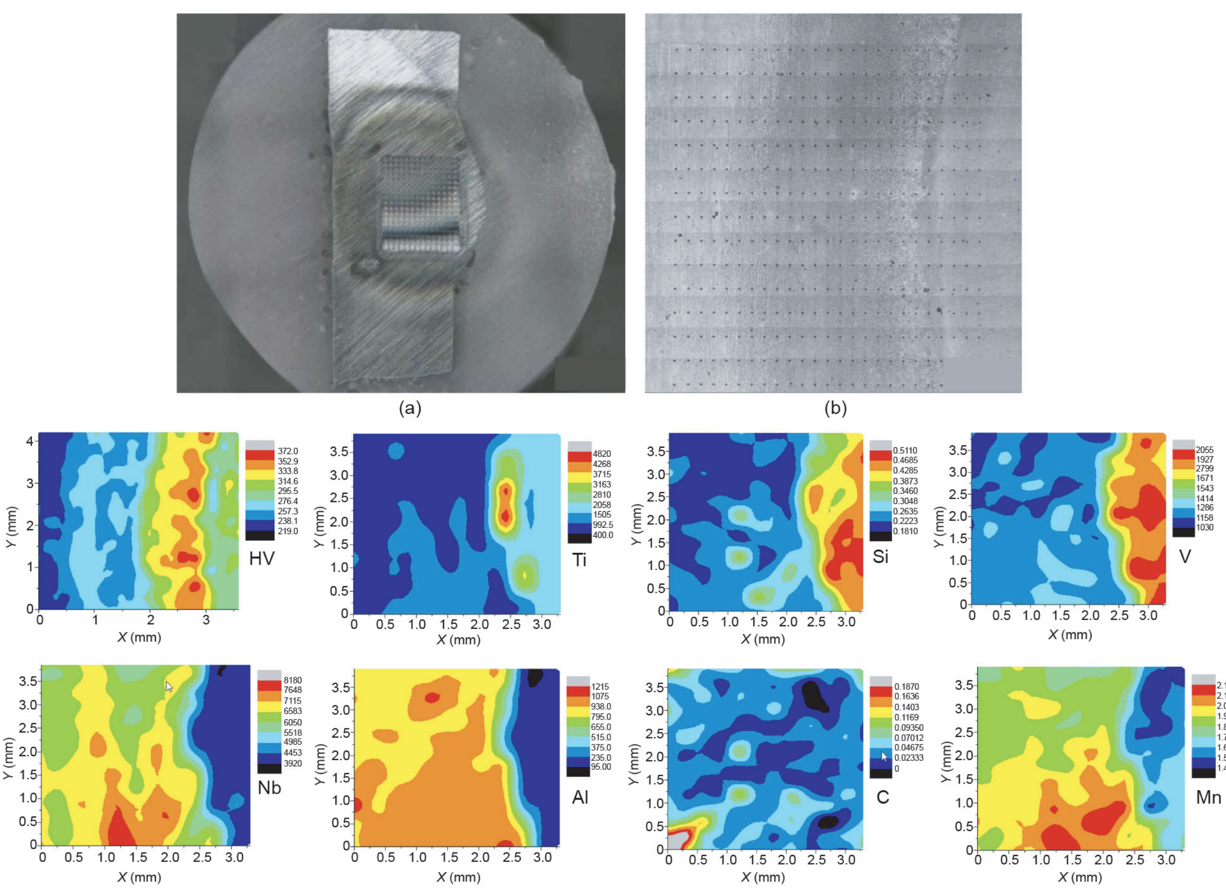


图7. X80管线钢堆焊区激光原位表征与显微组织和维氏硬度分布研究。(a) X80管线钢堆焊区扫描面积3.3 mm×3.9 mm;(b) 全视场显微组织及维氏硬度分布;(c) 堆焊区维氏硬度分布与成分分布图。

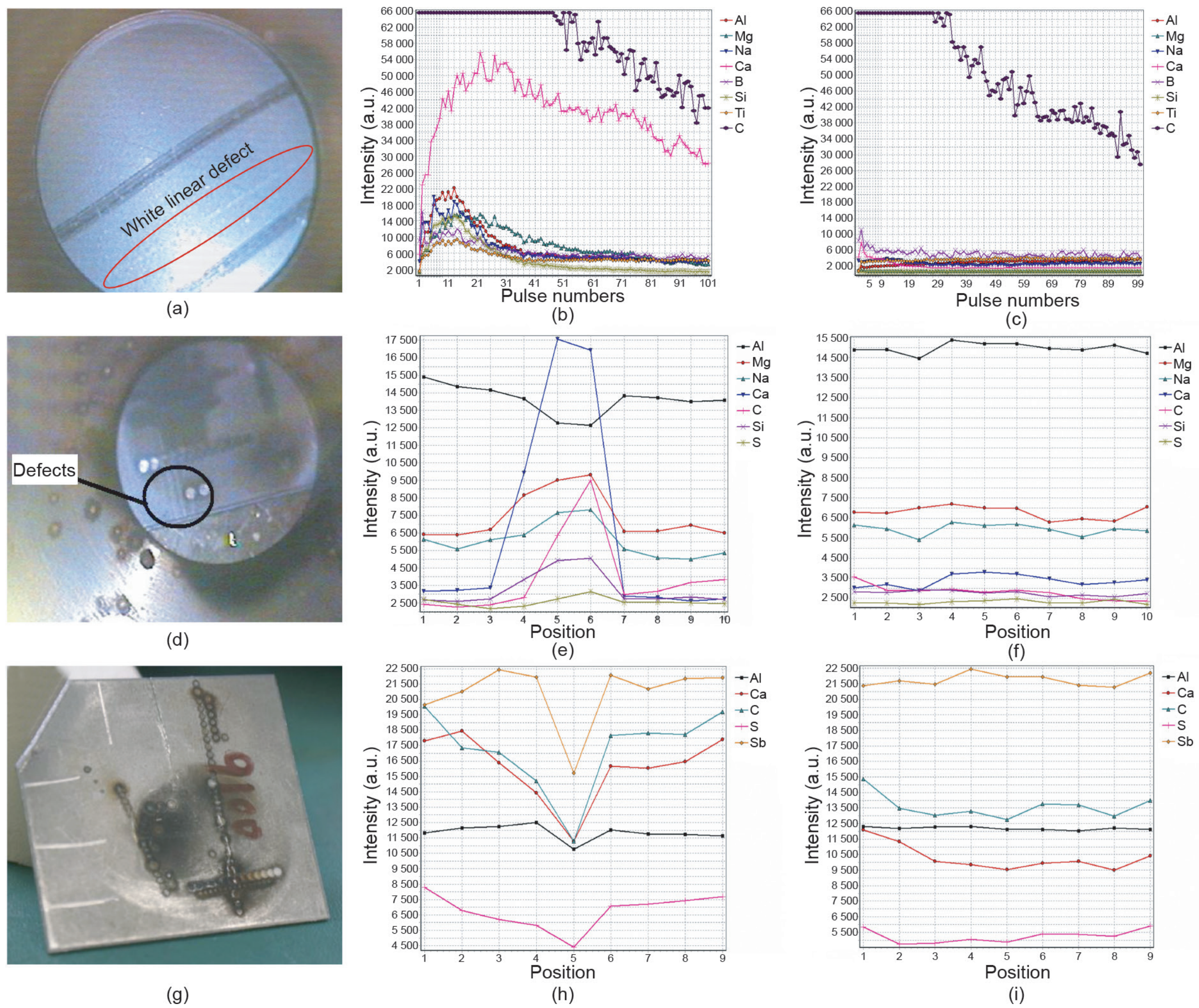


图8. 激光原位在汽车板缺陷的深度及线扫描研究。(a) 镀锌板线状缺陷;(b) 缺陷处深度分析;(c) 非缺陷处深度分析;(d) 汽车板带状缺陷;(e) 缺陷处表面线扫描;(f) 非缺陷处表面线扫描;(g) 汽车板划痕缺陷与人工划痕;(h) 划痕缺陷处线扫描;(i) 人工划痕处线扫描。

性和偶然性等问题，使得金相组织表征更具全面性。王海舟等[153]采用全视场金相原位技术对铁硅合金中的马氏体和铁素体进行了统计分布分析，结果表明，组织结构的灰度值与碳含量、硅含量、碳硅含量比及维氏硬度具有量化相关性（图15）。

2.3.6. 大尺寸跨尺度高通量扫描电镜原位统计分布表征技术

电镜原位技术采用自主设计的高亮度场发射电子源、高分辨率电磁复合物镜、直接电子探测器等技术，实现对大尺寸样品组织图谱的高通量获取，同等质量的图像其拍照时间是传统扫描电镜的1/50。智能软件集成多种特定材料的专业图谱库，全自动获取并标定组织结构类别及特性，采用图形处理单元（graphic processor unit, GPU）多线程并行运算及大数据挖掘，更全面地统计解析大尺寸材料的整体组织结构的分布情况，更利于与材料的成分和性能分布建立统计映射相关性。王海舟等[154]采用电镜原位技术对直径12 mm的镍基单晶高温合金样品进行表征，获取了全表面γ′相的分布信息，见图16。对数据进行原位统计解析，结果显示小尺寸γ′相主要分布在枝晶干，大尺寸γ′相主要分布在枝晶间，见图17。

2.3.7. 流体微探针应力应变原位统计分布表征技术

流体微探针原位技术基于等静压原理，当样品表面在高压流体（气体或液体）作用下，由于样品具有非均匀性的本质，不同组织结构的位置将产生不同的形变，通过建立每个位置上的微小形变与组织结构的相关性，实现应力应变在原始位置上的统计分布表征。流体可视为连续分布的且压力均匀的微探针，因此流体微探针原

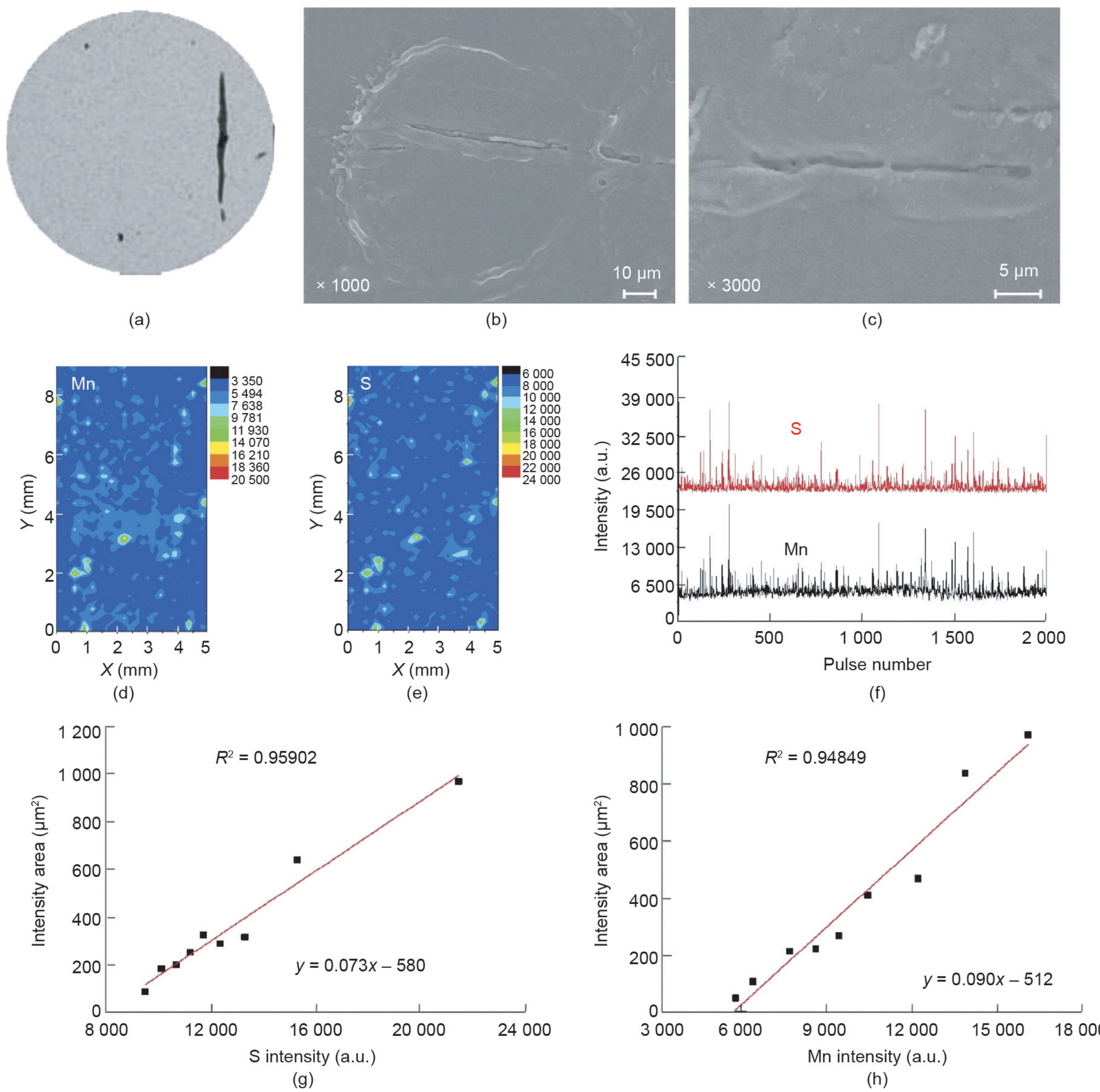


图9. 激光原位技术在MnS夹杂物上的研究。(a) MnS夹杂物；(b) 夹杂物部分烧损；(c) 夹杂物完全烧损；(d) Mn强度分布图；(e) S强度分布图；(f) S和Mn强度合成图；(g) S强度与夹杂物面积的线性拟合；(h) Mn强度与夹杂物面积的线性拟合。

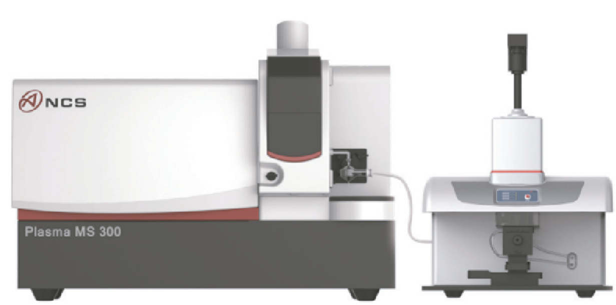


图10. 激光剥蚀电感耦合等离子体质谱原位分析系统。

位技术是一种纳米—微米—毫米—厘米真正意义上的连续跨尺度、高通量力学性能表征技术。冯光等[155]采用流体微探针原位技术对高铬白口铸铁的样品表面形变与组织结构分布以及维氏硬度分布进行了研究，结果表明，形变与弹性模量、等效模量和硬度有紧密相关性，见图18。

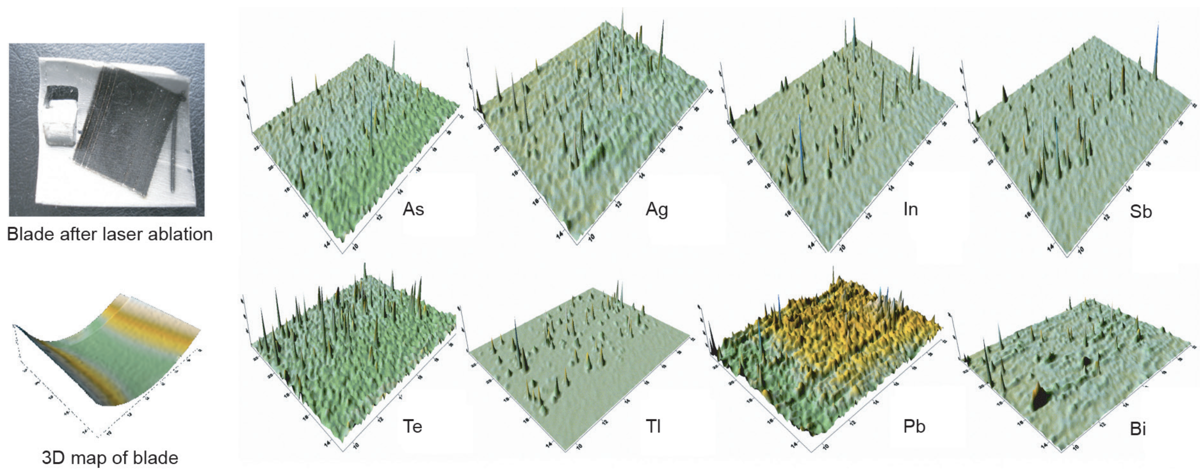


图11. 定向凝固高温合金涡轮叶片多晶带析出低熔点元素的分布表征。

图12. 微束荧光原位分析仪原型机。

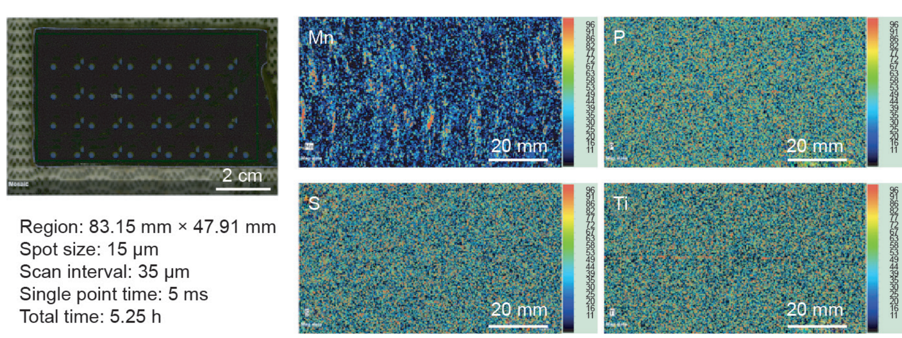
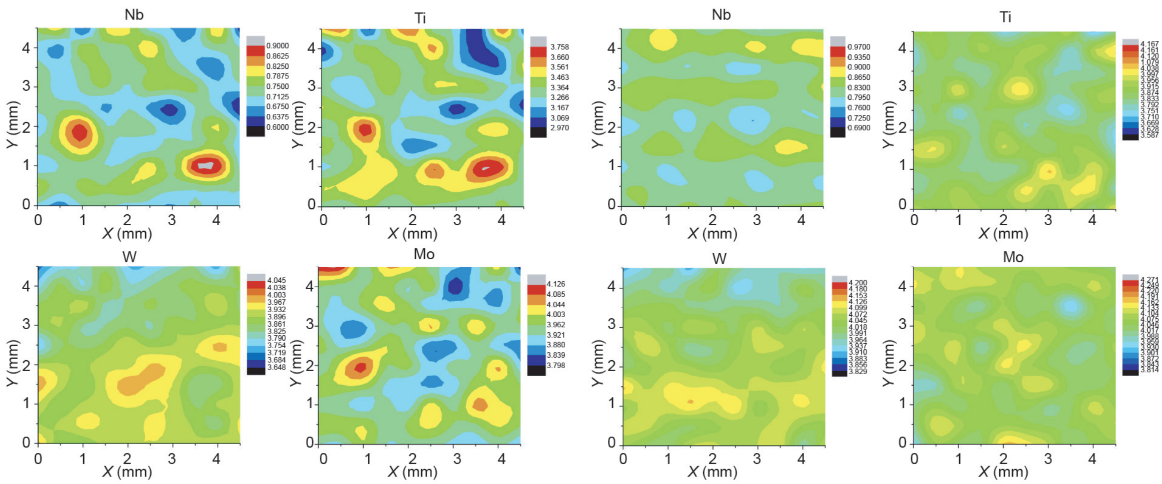


图13. 耐候钢薄板坯微区成分偏析统计分布表征。

3. 高通量统计映射表征技术的示范应用

3.1. 超超临界机组用 G115 耐热钢时效过程中 Cu 的统计映射表征

超超临界燃煤发电是实现节能减排的重要举措，耐热材料的研发是制约先进超超临界火电机组发展的最大瓶颈。G115马氏体耐热钢是基于现有质量分数为9%~12%的Cr耐热钢开发的，其极限服役温度可突破至650 ℃，具有重大的工程意义。G115钢在合金设计时通过添加质量分数为1.0%的Cu起析出强化作用。然



Element (casting)	Average content (%)	RSD (%)	Maximum segregation degree
Nb	0.728	7.71	1.24
Ti	3.346	5.00	1.12
Mo	3.951	1.60	1.04
W	3.882	1.52	1.03

Element (heat treatment)	Average content (%)	RSD (%)	Maximum segregation degree
Nb	0.824	3.37	1.08
Ti	3.873	2.01	1.05
Mo	4.066	1.12	1.03
W	4.034	1.32	1.02

(c)

图14. 不同工艺高温合金中Nb、Ti、Mo、W成分统计分布表征。(a）铸态;(b）热处理态;(c）成分统计分布对比。

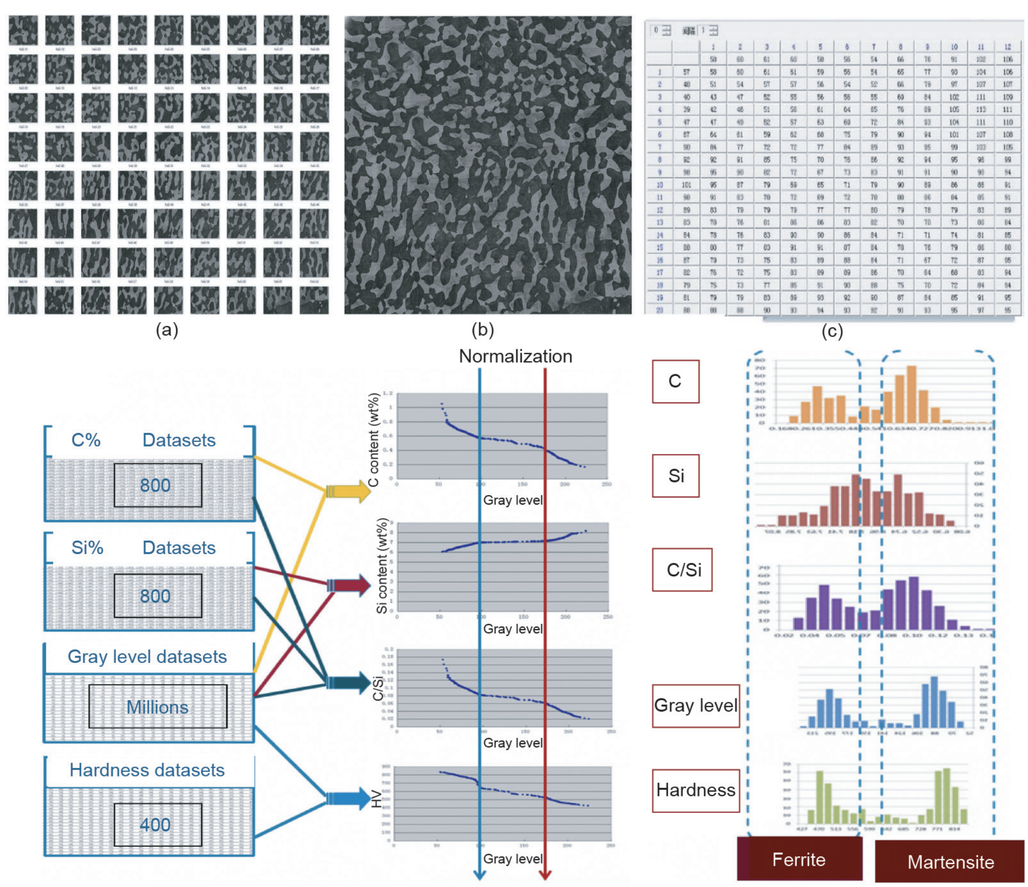


图15. 全视场金相原位技术在铁硅合金中的统计分布表征。(a）72个视场;(b）全视场拼接图谱;(c）全视场数字信号;(d）成分-灰度值-组织结构-硬度的统计频度峰具有良好相关性。

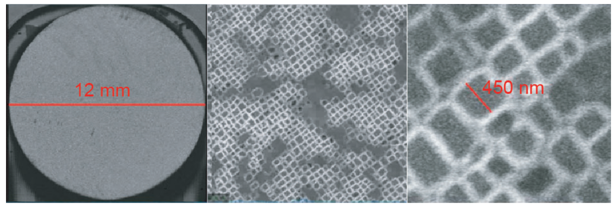


图16. 单晶高温合金全视场及放大后的γ′相。

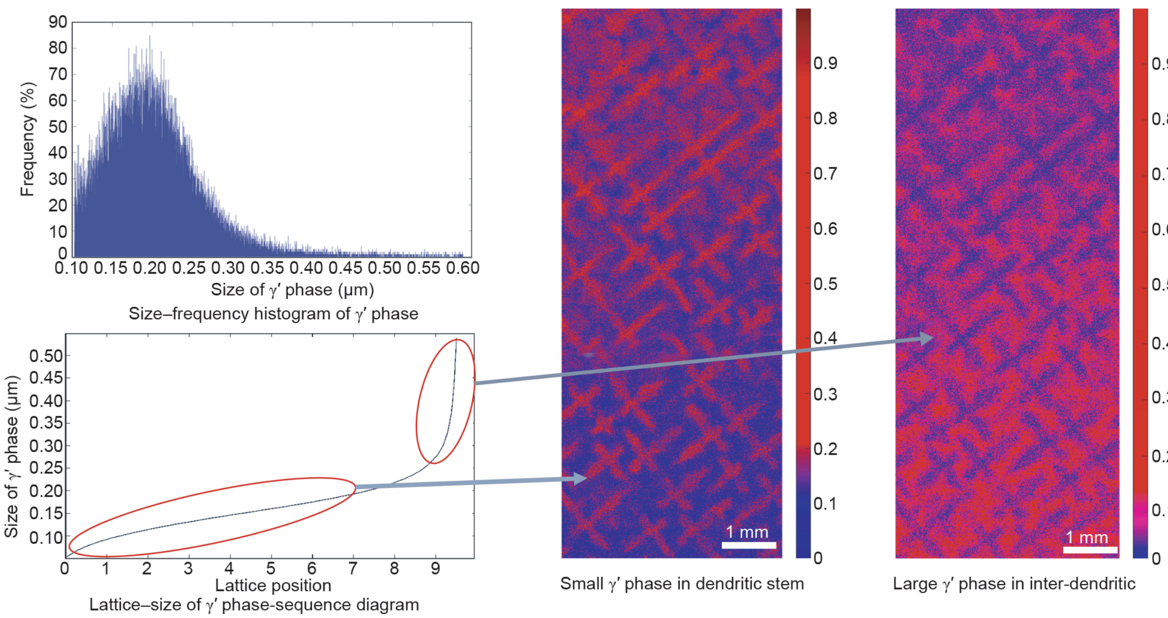


图17. 不同尺寸γ′相的分布情况。

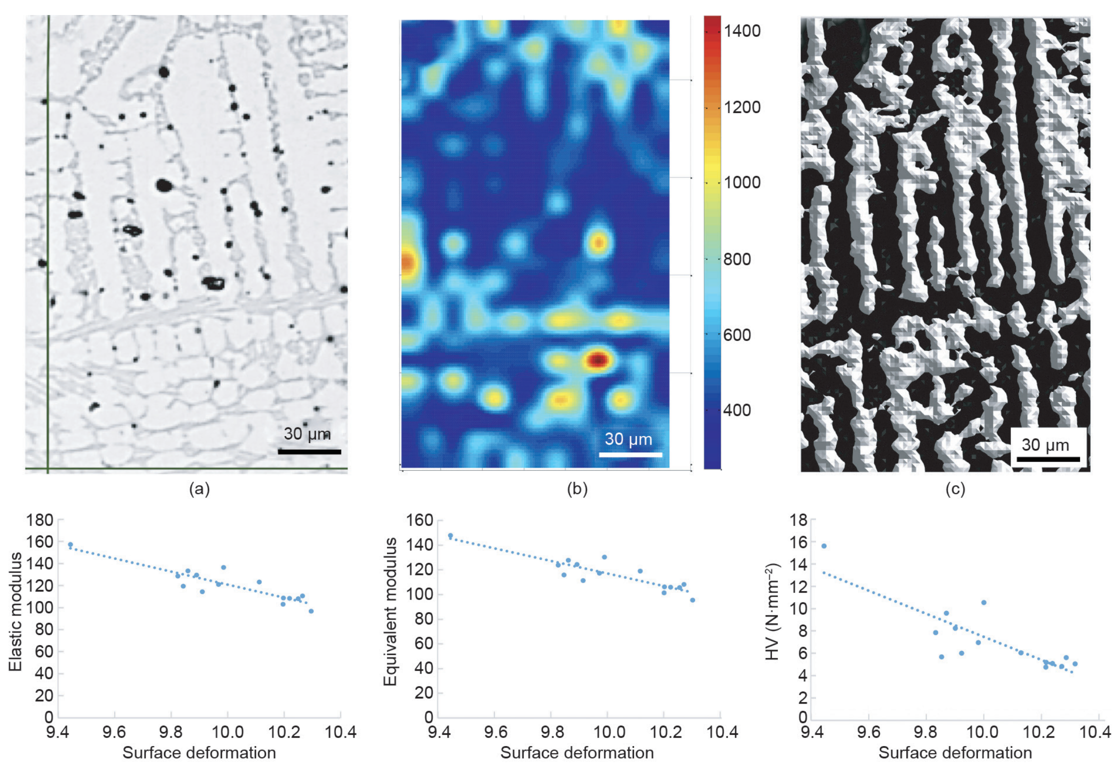


图18. 流体微探针原位技术在高铬白口铸铁中的研究。（a）金相组织图；（b）维氏硬度分布图；（c）等静压形变分布图；（d）弹性模量与形变相关性；（e）等效模量与形变相关性；（f）维氏硬度与形变相关性。

而，由于马氏体钢中Cu析出相表征困难，G115钢中Cu的存在形式、分布状态及作用机理等尚不清楚。杨丽霞等[156]采用微束荧光原位技术对试样全表面尺寸（8.1 mm × 8.1 mm）进行介观至宏观跨尺度表征，由二维强度分布图可以看出，各元素在介观状态呈均匀分布，没有明显偏析，说明微束荧光的分辨率不足以表征样品中Cu的差异性（图19）；随后，采用扫描电镜能谱（SEM-energy dispersive spectroscopy, SEM-EDS）对微束荧光分析结果中强度最高的区域进行定位，并进行微观至介观跨尺度表征，在1000× 放大倍数时由于空间分辨率较低，表征区域内（300 μm× 300 μm）各元素仍呈较均匀的弥散分布状态（图20）；进而，选取图中Cu稍显富集的白色方框区域在20 000× 下再次微观表征，此时，在15 μm× 15 μm区域内可快速筛选出含Cu特性基本单元，能谱面分布图中Cu富集区域分布于界面或晶界，且其他元素均呈负偏析，表明Cu以富Cu颗粒状态单独存在于界面或晶界，而不与其他元素成相（图21）；为进一步确定富Cu颗粒的存在形式，采用扫描透射电镜（scanning transmission electron microscopy, STEM）对试样薄区界面区域（3 μm× 3 μm）进行多视场面扫描微观表征，结果显示：G115钢中富Cu颗粒为面心立方结构（face-centered cubic structure, FCC）的富Cu相，其Cu的质量分数约为90.28%（表1），呈椭圆形或球形，等效直径为50~242 nm，平均直径为114 nm，常与$M_{23}C_6$、Laves相沿板条界共生，也可独立存在于板条界处，周围往往分布有大量位错（图22）；另外，采用三维原子探针（three-dimensional atomic probe, 3DAP）对不同时效时间的G115钢基体中的Cu进行了表征，结果表明，时效时间的延长将促进Cu的析出（表2）。最终，采用高通量统计映射表征技术从宏观材料至微观特征区域逐级定位筛查，实现含Cu特性基本单元的存在形式及分布状态等精细表征，并通过对G115钢时效过程中Cu的系统表征揭示了Cu的演变规律（图23）。

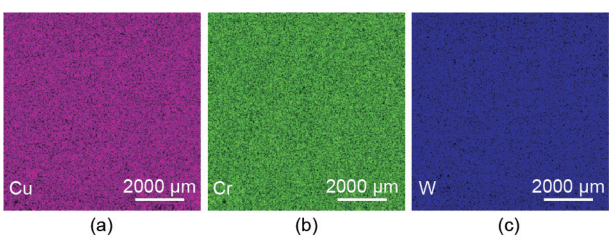


图19. 微束荧光原位技术介观至宏观跨尺度分布表征。（a）Cu的μ-XRF分布；（b）Cr的μ-XRF分布；（c）W的μ-XRF分布。

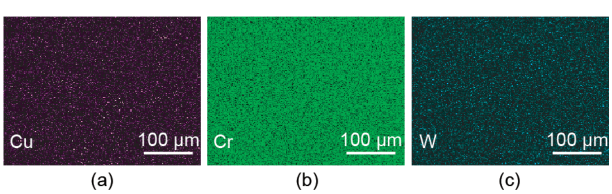


图20. EDS-SEM 1000×下微观至介观跨尺度分布表征。（a）Cu的SEM分布；（b）Cr的SEM分布；（c）W的SEM分布。

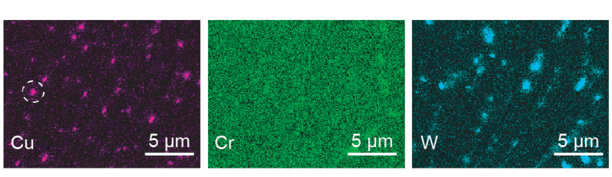


图21. EDS-SEM 20 000×下微观至介观跨尺度分布表征。（a）Cu的SEM分布；（b）Cr的SEM分布；（c）W的SEM分布。

3.2. 大尺寸变形FGH96涡轮盘成分结构和性能统计映射表征

镍基高温合金是飞机发动机和燃气轮机涡轮盘的关

表1 G115钢中含Cu特性单元的STEM-EDS分析

Item	Cu	Fe	Cr	Co	Mn
Content (wt%)	90.28	5.64	2.13	0.50	1.44

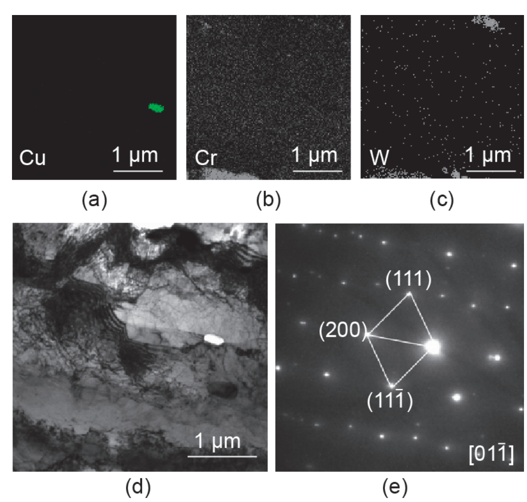


图22. STEM的微观分布表征。（a）Cu的STEM分布；（b）Cr的STEM分布；（c）W的STEM分布；（d）Cu的透射电镜（transmission electron microscopy, TEM）图；（e）Cu的选区电子衍射谱。

表2 G115钢时效过程基体中Cu含量的3DAP分析结果

Item	Cu content in matrix
Tempered	0.48 ± 0.20
650 °C heat treated (equivalent 3000 h)	0.18 ± 0.05
650 °C heat treated (equivalent 8000 h)	0.15 ± 0.03

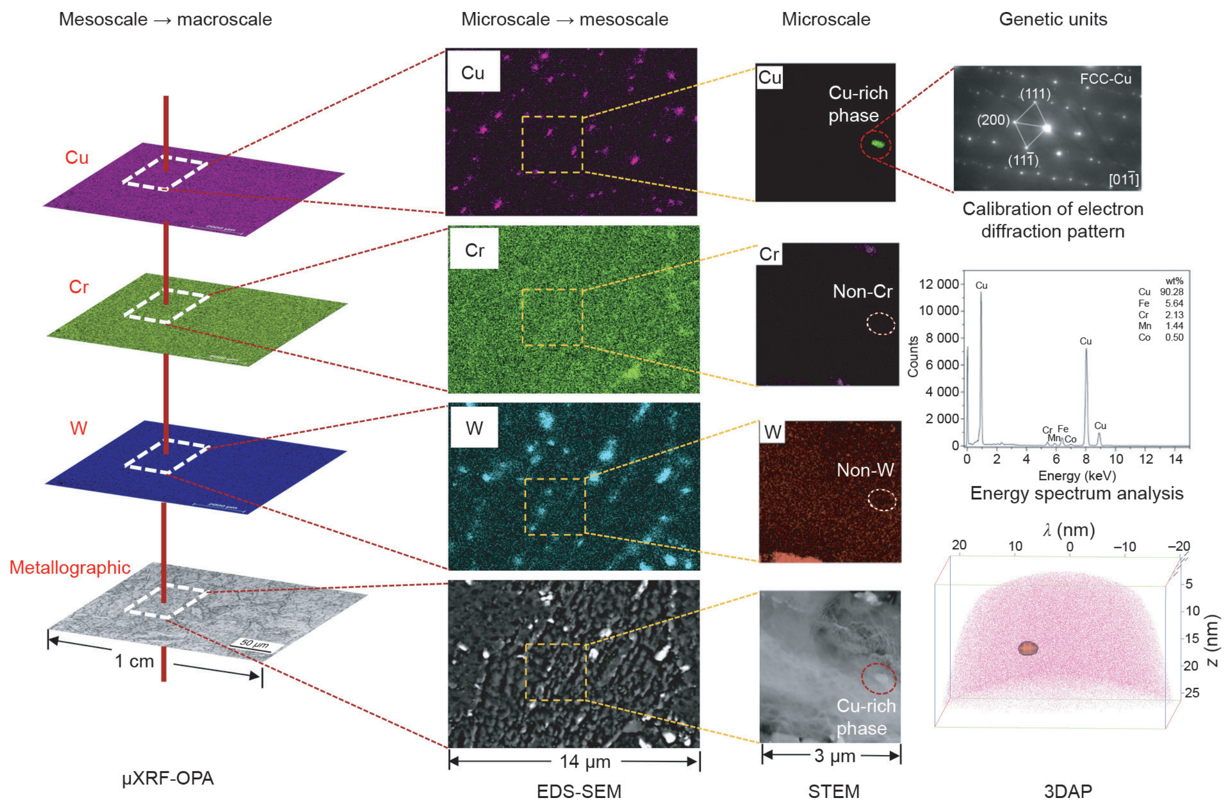


图23. G115钢中富Cu相的跨尺度高通量统计映射表征。

键材料，由于化学成分复杂，服役环境恶劣，对性能要求严格，导致其研发改性周期长。大尺寸变形FGH96涡轮盘是采用电渣重熔连续定向凝固（electroslag remelting continuous directional solidification, ESR-CDS）工艺制备铸锭，多向锻造和等温锻造成型，其各项力学性能与粉末FGH96合金相当，该变形合金尚处于工程化研制阶段。卢毓华等[157,158]采用火花原位、全视场金相、扫描电镜等多种统计分布表征技术对大尺寸变形FGH96涡轮盘切片进行了表征，分别获取了多种成分、γ′相总量、一次γ′相、二次γ′相、三次γ′相、γ′相粒径、晶粒度、碳化物相、维氏硬度、室温拉伸以及高温蠕变等多个参量在盘片上分布数据（图24），并且将这些数据建立了位置一一对应的统计映射关系，结果发现，0~100 nm范围内γ′相的相对质量分数以及进入γ′相的Co、Mo的原子分数对高温蠕变性能具有重要影响，构建了高温合金基本单元γ′相和高温蠕变性能间的区域统计映射相关性数学模型（图25），对于高温合金涡轮盘的改性具有重要指导作用。

4. 展望

高通量统计映射表征技术是基于原位统计分布分析方法发展而来的一种表征新方法，目前已发展出火花原位技术、激光原位技术、激光剥蚀原位技术、微束荧光原位技术、全视场金相原位技术、电镜原位技术、流体微探针原位技术等一系列新方法和具有自主知识产权的新装置。高通量统计映射表征技术在“自然芯片”解析表征上取得了大量的应用成果，实现了各种碳钢、不锈钢、有色金属、连铸板坯、镀层板、高温合金、耐热钢的成分、组织结构和力学性能的跨尺度高通量表征，通过从宏观到微观的逐级分析，筛查出影响材料性能的基本单元（组），建立了成分-组织结构-性能的跨尺度统计映射模型，其最大优点是与实际生产工艺相近，有利于指导材料的改性和工艺优化，其缺点是设计调控自由度受工艺限制。后续将进一步开展数据分析研究，对这些从宏观至微观特征区域逐级筛查出的数据进行人工智能学习，再从微观反演重构为整体宏观材料在每个位置及微区上的精细组成、结构和性能，从而实现材料的反演重构和按需逆向设计，加速新材料的发现。

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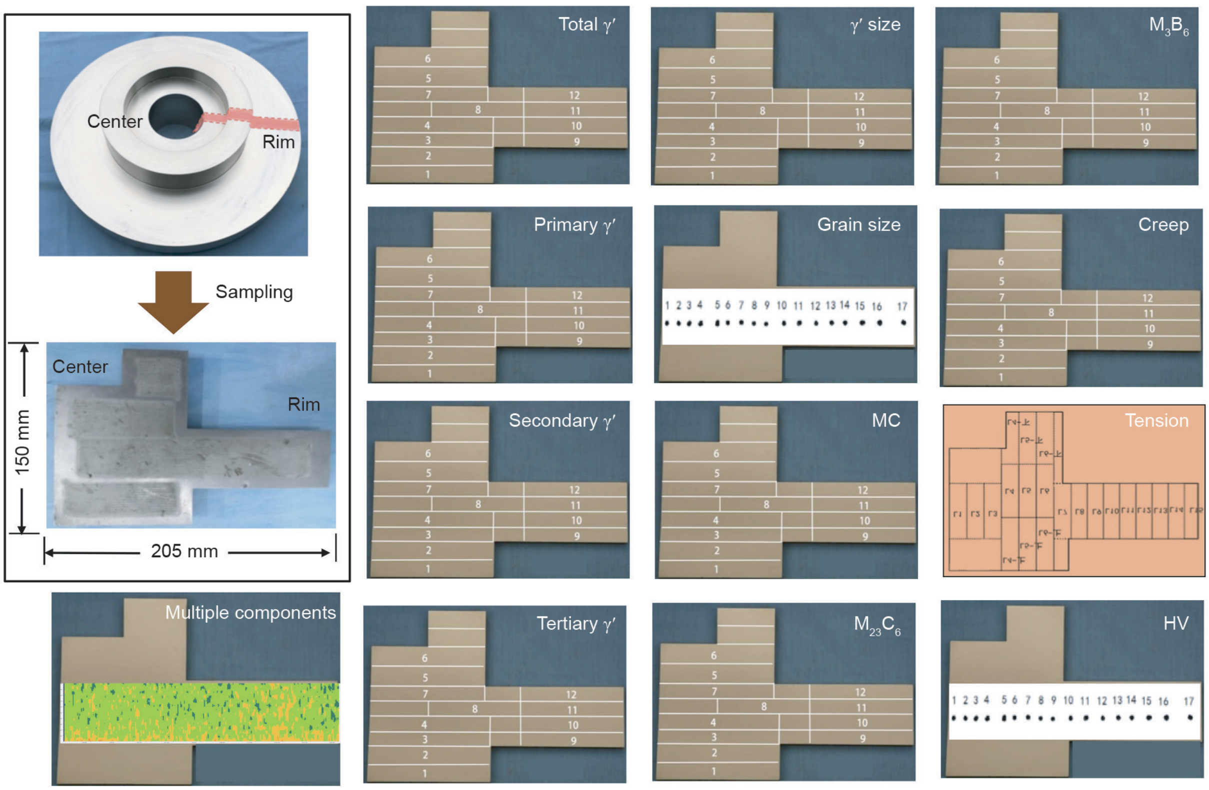

图24. 多参量高通量统计分布表征。MC、$M_{23}C_6$、M_3B_6型碳化物。

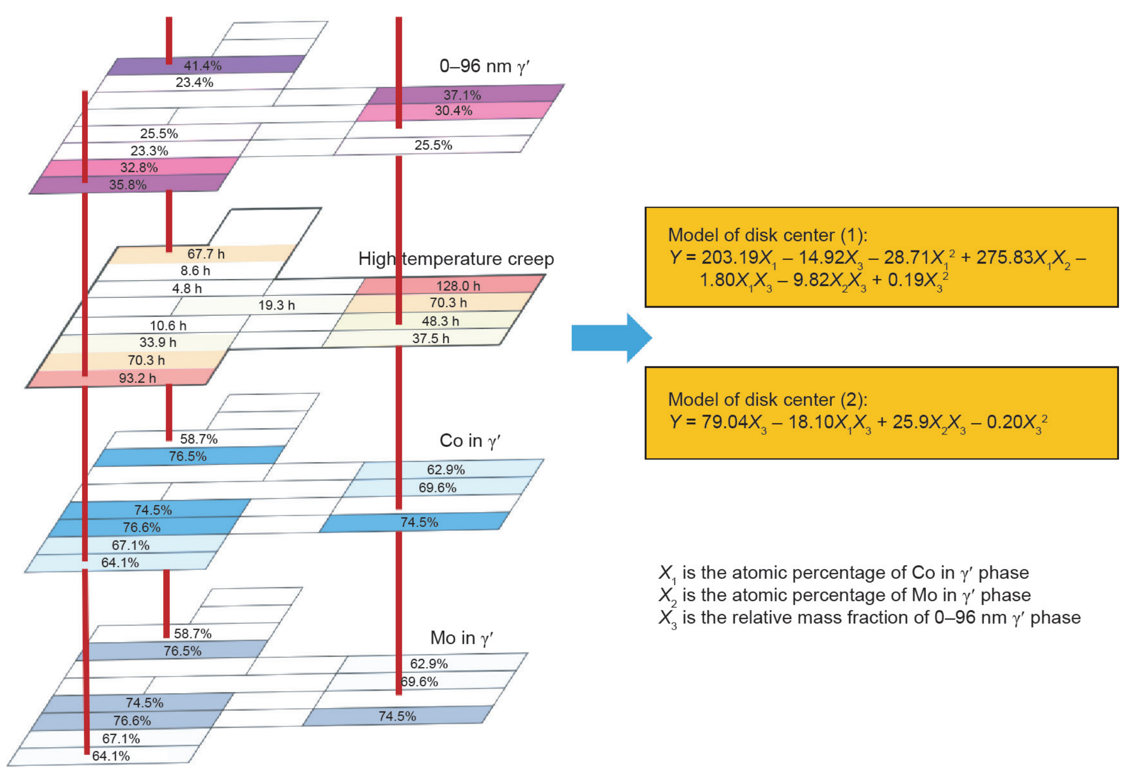

图25. γ′相与高温蠕变统计映射相关性数学模型。

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Compliance with ethics guidelines

Haizhou Wang, Lei Zhao, Yunhai Jia, Dongling Li, Lixia Yang, Yuhua Lu, Guang Feng, and Weihao Wan declare that they have no conflicts of interest or financial conflicts to disclose.

Appendix A. Supplementary data

Supplementary data associated with this article can be found in the online at https://doi.org/10.1016/j.eng.2020.05.005.

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